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**STUDIES ON SELECTIVE FLOCCULATION OF
HIGH-ALUMINA INDIAN IRON ORE SLIME**

By

AJAY BALDAWA

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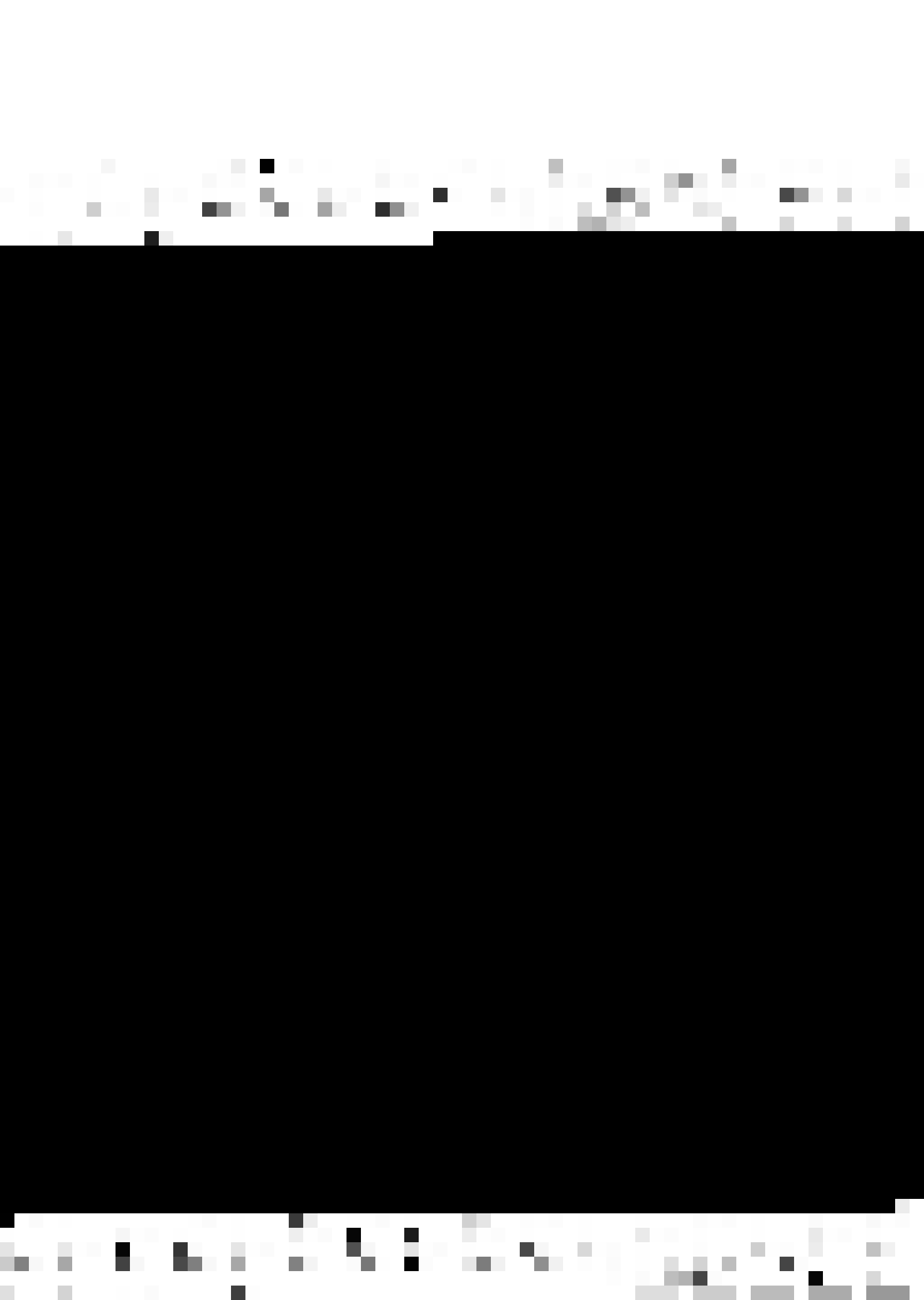
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DEPARTMENT OF METALLURGICAL ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY, KANPUR

JULY, 1980



STUDIES ON SELECTIVE FLOCCULATION OF HIGH-ALUMINA INDIAN IRON ORE SLIME

A Thesis Submitted
In Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

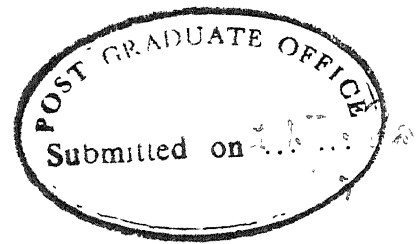
By
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to the
DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
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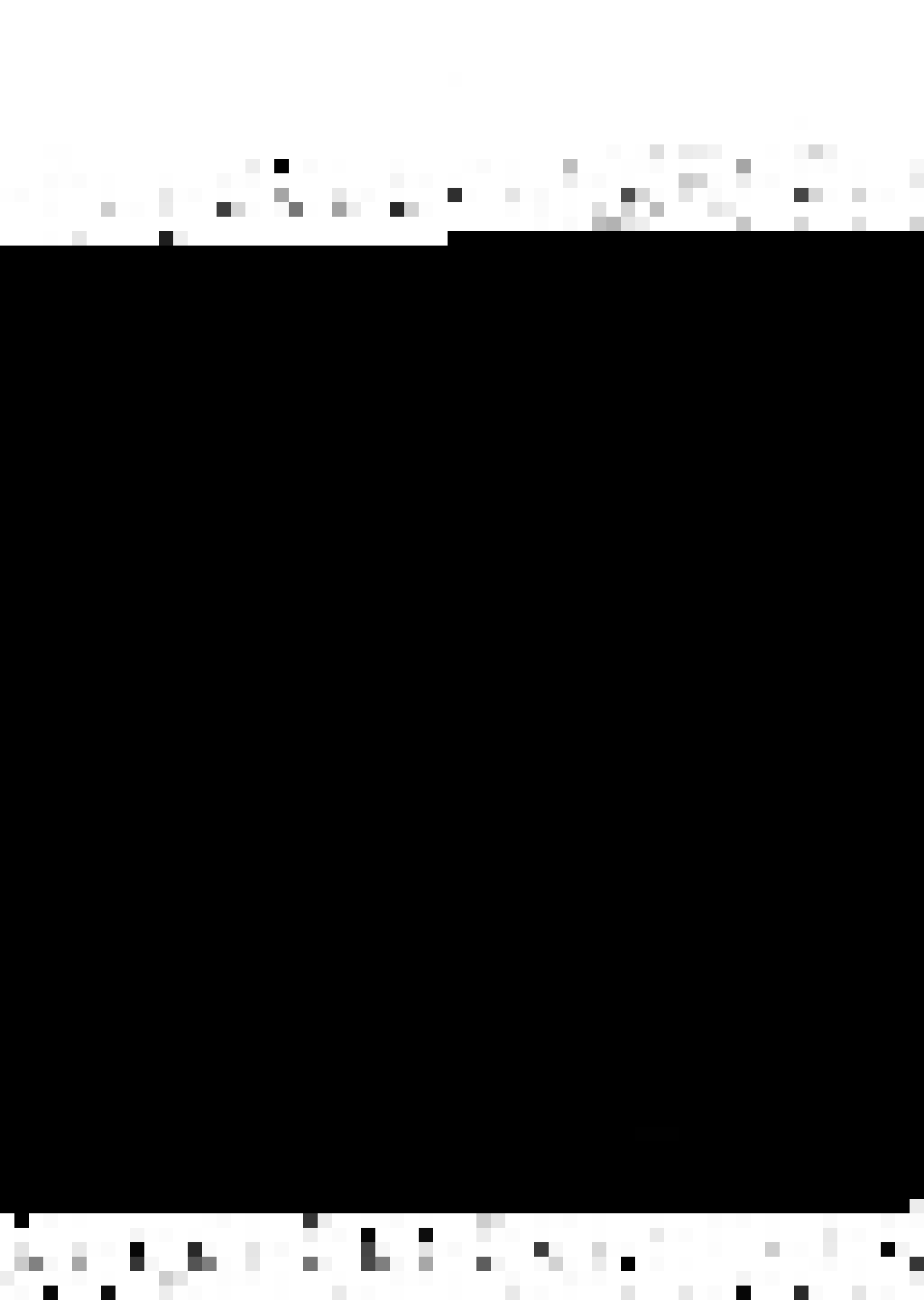


CERTIFICATE

Certified that the present work entitled 'Studies on Selective Flocculation of High- alumina Indian Iron Ore Slimes' by Ajay Baldawa has been carried out under my supervision and has not been submitted elsewhere for the award of a degree.

Kanpur
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Ajay Baldawa
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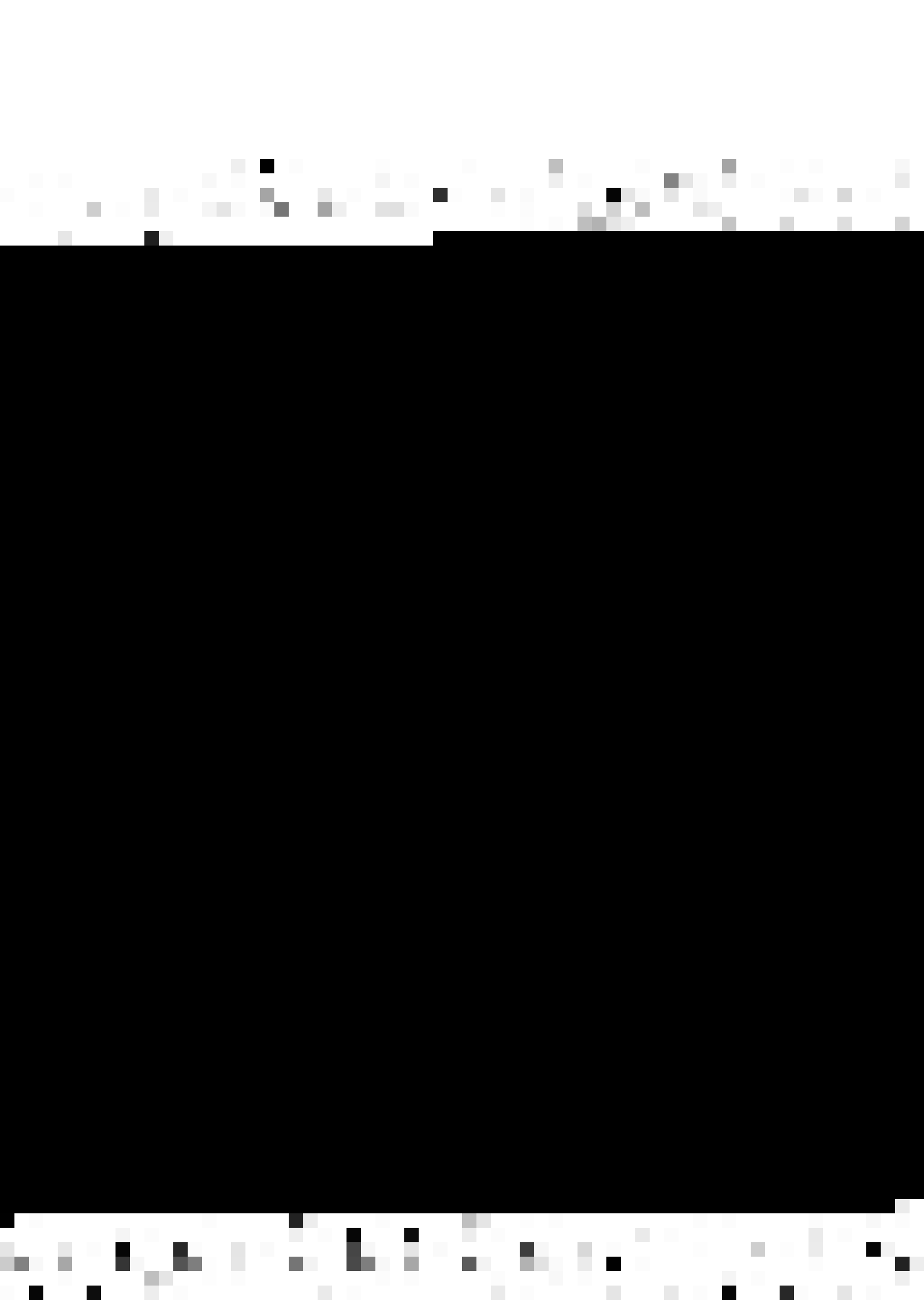


ABSTRACT

This work represents an effort towards beneficiation of a high-alumina iron ore slime (Barsua, Mourkela Steel plant) by selective flocculation. Liberation of the aluminium bearing minerals (and possibly of hematite also) is very difficult and occurs only at very fine size ranges around 2 μ m.

Studies have been made on sized fractions such as 1-8, 1-20 and 0-74 μ m Barsua slime (as well as limited experiments on 0-74 μ m Bhilai ore slime and mixtures of pure clay minerals and hematite). Flocculation experiments were done with different concentrations of starch, starch phosphate and various dispersants and modifiers. Other variables studied were pH, agitational speed, conditioning time, pulp concentration, number of stages in multiple-stage flocculation etc. Some flocculation experiments were done using cationic flocculant, cationic collector, kerosene droplet etc.

In each case, selectivity index, appropriately defined in the text, was calculated and taken as a measure of separability. Some of the best results obtained such as



82.8 pct. Grade, 81 pct. Recovery, 2.66 s.i. (Fig. 4, Table 7) and 83 pct. Grade 21 pct. Recovery, 1.53 s.i. (Fig. 5, Table 8) are still not good enough.

The reasons for unsatisfactory separation have been identified as poor liberation, problems connected with ultra-fine particles, possible coating or hetero-coagulation effect with clay etc. It has been suggested that further work may be done with mixtures of fine particles of pure minerals as well as on other iron ore slimes in which sufficient liberation has been achieved.

CHAPTER I

INTRODUCTION

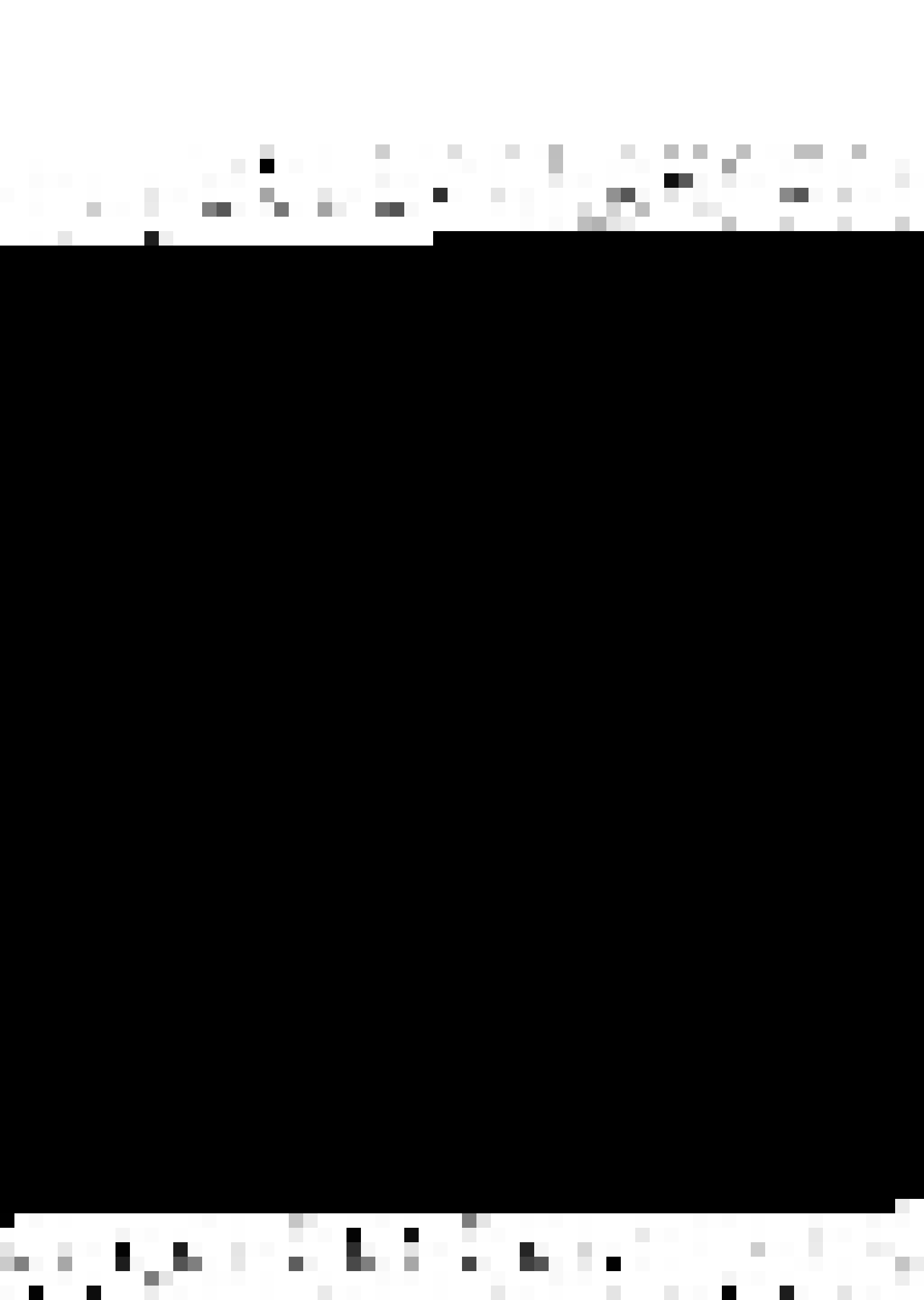
India is fortunate in having large reserves of iron ore, the basic raw material for steel. The reserves are reported to be around 21,400 million tonnes with an average iron content of 52 pct. and above¹. A substantial amount of this reserve is hematitic and high grade in terms of iron content. However the problem with Indian iron ores is two-fold. One is the high content of the aluminium bearing minerals, the other being the relatively soft nature of the ore which generates considerable proportion of fines during mining and handling.

The Aluminium bearing minerals in the Indian iron ore give rise to large amount of Al_2O_3 in the iron Blast furnace. Alumina increases the viscosity of the Blast furnace slag as well as the Al_2O_3/SiO_2 ratio which should be low for efficient blast furnace operation. To counteract these factors, lot of dolomite stone and quartzite have to be added. This in turn results in an increase in coke rate, contamination level and silicon in the hot metal. Attempts to wash Indian iron ores for the purpose of removal of alumina have met with only limited success²⁻⁵. Although Al_2O_3/Fe ratio could be slightly reduced

from 0.11 to 0.095, the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ratio was actually increased from 1.66 to 1.84. Quartz, the predominant gangue material could be easily removed by washing but not the clay material containing aluminium.

Indian Iron ore is of relatively soft nature, and the introduction of mechanisation and heavy blasting of the mines is generating larger quantities of fines^{2,3,6,7}. The proportion of -10 mm fines from Indian mines is never less than 35 pct and is likely to increase when mining is carried out at greater depths ~~area~~. These fines are of inferior quality (contain increased percentage of Silica and alumina), for smelting as such in the blast furnace. Moreover they can not be treated in the blast furnace which requires close sizing of the lump ores. There are also large deposits of blue dusts or the natural fines in the lower layers of the deposits. As these fines can not be rejected, they require beneficiation prior to pelletisation or sintering to produce agglomerates which are suitable as blast furnace burden material.

The ultrafine particles respond poorly to conventional physical separation techniques like gravity separation, froth floatation etc.⁸ One process that appears promising for the fine particle separation is selective flocculation. Ideally this should involve aggregation of desired mineral species



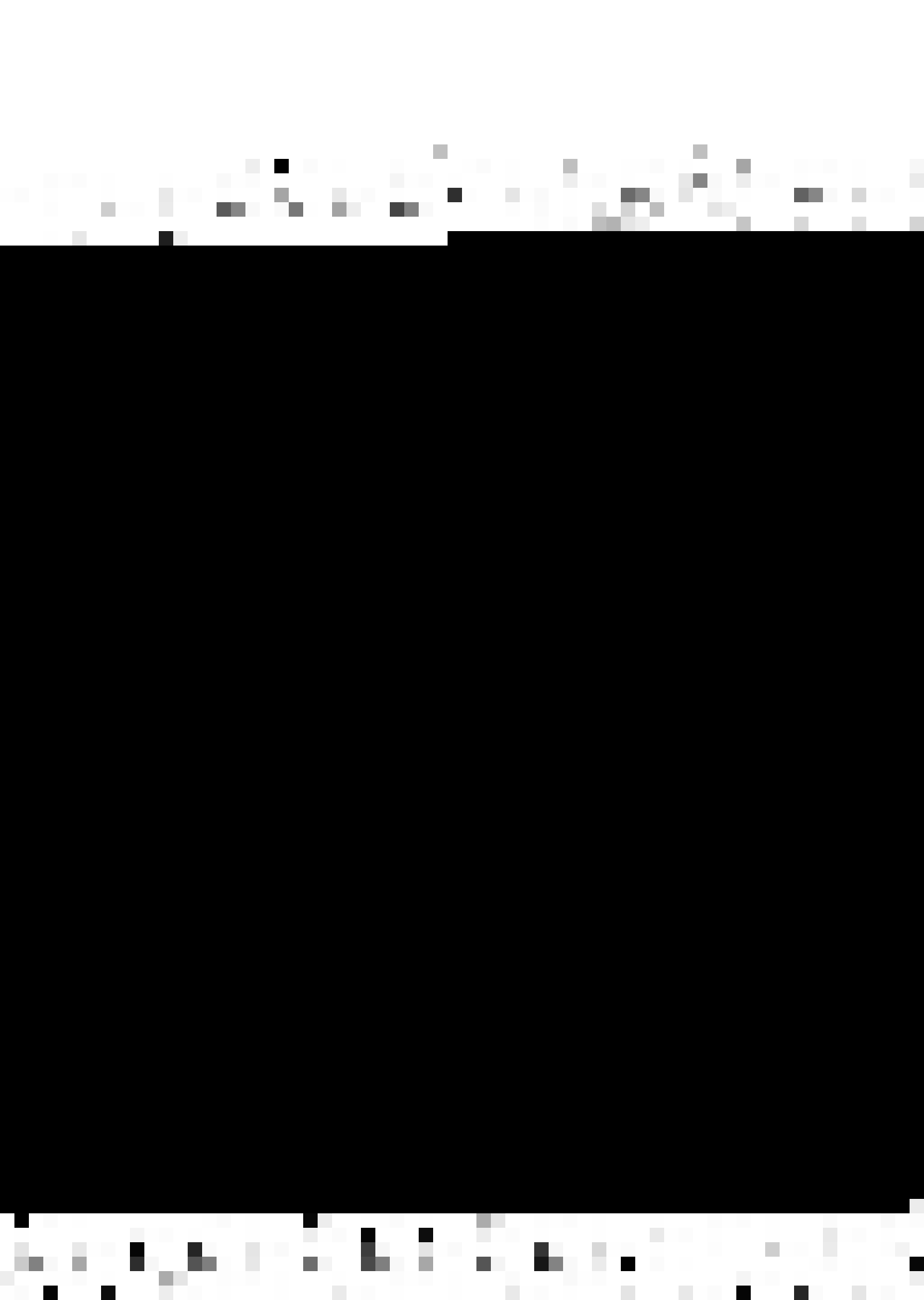
into flocs leaving the other particulate species in suspension. Separation of the flocculated material by such processes as floatation, sedimentation or elutriation should result in the desired concentration^{9,10}. Success of the technique of separation by selective flocculation depends on the liberation of particulates from each other, preferential adsorption of flocculants on particles and bridging of these into flocs by the adsorbed polymer molecules, and effective separation of the flocculated mass from the suspension using a technique that will not produce redispersion of flocs.

Fine particles constituting a mineral mixture usually have a tendency to coat each other and, thus, the selectivity in flocculating a particular mineral is significantly lowered⁸. This problem of slime coating and heterocoagulation is usually overcome by use of dispersants which prevent inter-particle adhesion. At least one component should be well dispersed⁸. The second requirement, and the essence of the process, is that a flocculant selectively adsorbs on only one of the constituents of the mixture. Selective flocculation then follows, after which the flocs of one component can be removed from a dispersion of the other mineral.

Forces responsible for adsorption of polymers result mainly from three types of bonding, namely, electrostatic,

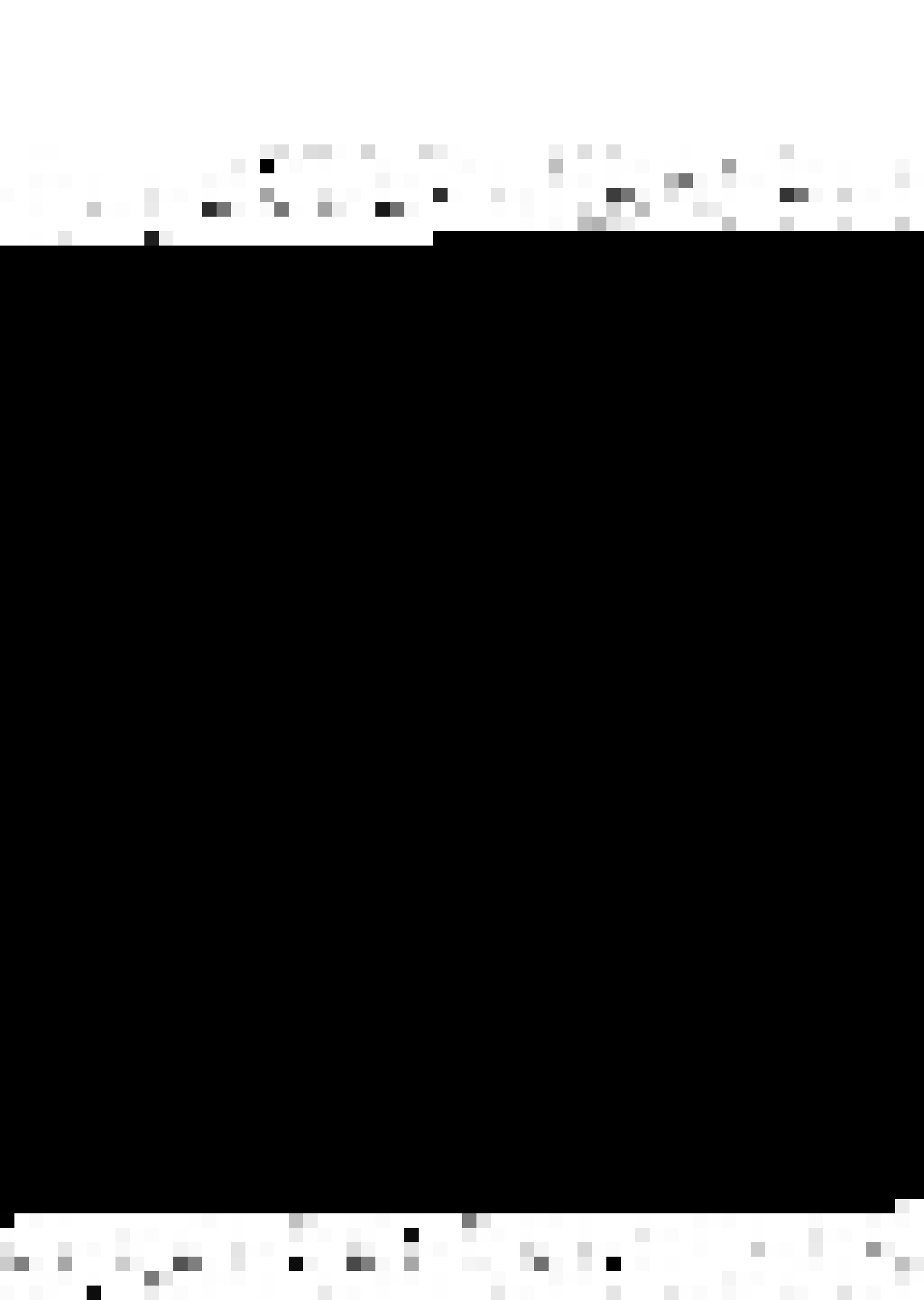
hydrogen and covalent bonding¹⁰. The predominance of any of the above mechanisms over the other depends on the particular mineral/polymer system and the properties of the aqueous medium. Under favourable conditions, more than one type of mechanism could be operative.

Reported work on selective flocculation has been mostly with binary mineral system⁸. Hematite-Quartz system has been studied by many workers using starch or Polyacrylamides as flocculant and sodium silicate or Phosphates of Sodium as dispersants⁸⁻¹¹. Beneficiation of multicomponent natural ores by selective flocculation has been attempted by Frommer et al¹², Hicks et al¹³, Iwasaki et al¹⁴ and Gururaj et al¹⁵. In few cases, success has been achieved in separation of multicomponent natural ores by selective flocculation¹⁰. Commercial development of Tilden's billion ton iron ore deposit is a major breakthrough in concentration technology and is the first commercial application of this technique^{16,17}. Starch and Sodium Silicate have been used as flocculant and dispersant respectively for the beneficiation of Tilden Mine ore which is ground to 85 pct. minus 500 mesh (25 microns)¹⁸. Tilden ore body has a grain size less than 25 microns, so the liberation of minerals would occur only at very low particle sizes.



Barsua and Bhilai Iron ore which represent very typical Indian cases are also fine grained ore rich in Alumina¹⁵. X-ray, electron microscopic, electron diffraction, and electron probe micro-analysis experiments have established the size, elemental composition and mineralogical phases of alumina-rich grains¹⁵. This have been further confirmed by x-ray investigation of the leach residue after dissolution by Jackson's method¹⁹. These studies have indicated the presence of Illite, Kaolinite, Montmorillonite (also possibly Diaspore and Gibbsite) in Barsua ore and of Sillimanite, Illite, Aluminite, Hydrated Aluminium Silicate and Montmorillonite (and also possibly chloritoid, Kyainite, Corundum, and Kaolinite) in the Bhilai ore. The sizes of the aluminium containing grains varied between 0.03 to 2.0 microns for Barsua sample and 0.05 to 2.0 microns for the Bhilai sample.

The liberation studies²⁰ carried out by fractionating the Barsua ore sample into two fractions, namely Float and sink by Flutriation and Heavy Media Separation clearly showed that there was no significant liberation unless the particle sizes are below 20 micron and preferably below 15 micron. The X-ray studies of the 0-8 micron float, 8-15 micron, Float and 8-15 micron sink, after they were subjected to Jackson's CBD method of dissolution again substantiated poor liberation of Kaolinite and Montmorillonite (liberation of Illite was slightly better) as well as hematite in the 8-15 micron



size range²⁰ dissolved portion obtain by treating the fractionation by Jackson's CBD method¹⁹ was found to contain Al. As suggested by Jackson Al can be substituted in iron compounds and would be dissolved by CBD treatment. The fractionation data are reproduced here in Summary Table I. The data clearly showed that in the 0-8 micron size range although aluminium recovery ratio was around 34, iron recovery ratio was merely $\frac{1}{10}$. This considerable loss of iron in the float indicated severe locking with aluminium minerals. Of course, the electron microscopic measurements showed the aluminium containing mineral grains to be of micron sizes, but it was surprising to find that even the abundant hematite phase is difficult to liberate. It is obvious that very fine grinding is necessary for satisfactory liberation.

This work is in continuation of the previous work^{19,20}. The selective flocculation of 1-20 μ m and 1-8 micron Barsua slime has been tried. Commercial starch as well as starch phosphate (Phosphoryl group has been introduced in the starch) have been used as flocculant. The problem of flocculating fine particles has been highlighted. Some flocculation tests have been performed on 0-74 micron Barsua slime and Bhilai ore. And finally Flocc-Floatation tests on 1-8 micron Barsua slime and synthetic mixture of pure minerals have been performed.

1-1 SUMMARY TABLE I

Material Balance of -200 Mesh Larsua Slime Fractionated by Air
Flutriation and Heavy Media Separation

Size Frac- tion	Size Range in microns	Weight pct. %	Frac- tion	Weight pct. %	Assay in pct. Fe ₂ O ₃ SiO ₂ Al ₂ O ₃	pct. Fe ₂ O ₃	Recovery ⁹ Al ₂ O ₃	Selectivity Index
1	50-74	40.10	IF	35.58(88.72)	72.8 10.16 14.9	25.90	3.61 5.30	S.I. FeAl = 1.03 SI = 1.37
2	30-50	19.12	2F	14.97(78.31)	75.2 9.1 11.36	11.26	1.36 1.70	S.I. FeAl = 0.87
			2S	4.15(21.69)	76.8 5.7 15.38	3.19	1.24 0.64	S.I. FeSi = 1.28
3	20-30	19.14	3F	12.40(64.80)	73.6 9.8 12.4	9.13	1.22 1.54	S.I. FeAl = 0.97
			3S	6.74(35.30)	76.8 8.7 13.8	5.2	0.59 0.92	S.I. FeSi = 1.08
4	15-20	10.73	4F	4.62(43.05)	59.2 11.5 15.4	2.7	1.54 0.72	S.I. FeAl = 1.13
			4S	6.11(59.65)	70 10.15 14.2	4.28	0.62 0.86	S.I. FeSi = 1.16
5	8-15	6.09	5F	5.64(92.63)	57.6 16.2 20.4	4.5	0.91 1.16	S.I. FeAl = 1.36
			5S	0.45(7.37)	71.2 11.1 13.6	0.32	0.5 0.06	S.I. FeSi = 1.34
6	0-8	4.82	6F	4.33(89.75)	51.4 9.1 36.0	2.5	0.39 1.56	S.I. FeAl = 2.02
			6S	0.49(10.25)	53.6 15.8 9.2	0.26	0.08 0.046	S.I. FeSi = 0.77
F	Float	SI	Sink	Overall Slime Assay 71.1 pct. Fe ₂ O ₃ , 9.87 pct. SiO ₂ and 15.17 pct. Al ₂ O ₃				
9	Basis 100 units of Slime							



SUMMARY TABLE II

Selectivity Index values for 2-fraction cuts at different sizes
(Data from Summary Table I)

Size cut	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	S.I. Fe, Al	S.I. Fe, Si
50 mμ	29.37 ^φ (41.34)	3.87 (6.60)	5.96 (9.21)	1.05	1.05
80 mμ	43.82 (27.89)	5.47 (4.40)	8.30 (6.87)	1.14	1.12
20 mμ	58.15 (13.56)	7.28 (2.59)	10.76 (4.41)	1.33	1.24
15 mμ	65.13 (6.58)	8.44 (1.43)	12.34 (2.83)	1.51	1.3
8 mμ	68.95 (2.76)	9.40 (0.47)	13.56 (1.61)	1.72	1.12
Total	71.71	9.87	15.17		
Basis 100 weight units of -74 mμ material					

^φ Coarse fraction. Bracketted quantity refers to the remainder or lighter fraction.

CHAPTER II

MATERIALS AND PREPARATION

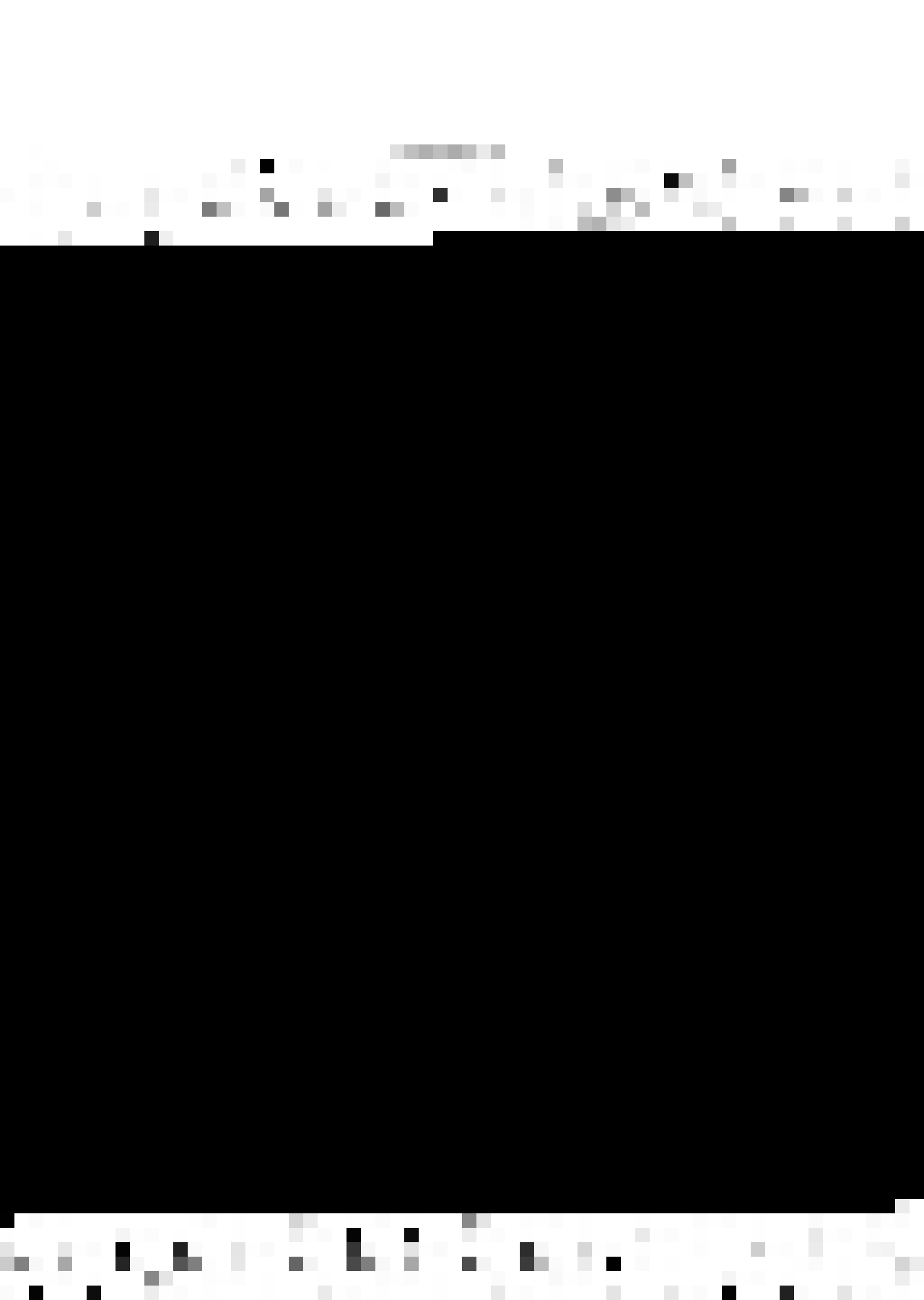
The details of the chemicals and the materials used for the present work are mentioned below.

2.1.1 Different Size Fractions of Barsua Slime and Zhilai Ore

(a) Barsua Slime:

The Barsua slime was supplied by Bourkela Steel Plant in two lots, lot I and lot II. The lot I was supplied in dry state with a particle size of $-74\text{ }\mu\text{m}$. The lot II was supplied in pulp form with presumatly ordinary water.

The different size fractions of Barsua slime were prepared by allowing the $0-74\text{ }\mu\text{m}$ size particles to settle in distilled water (pH adjusted to 9.0 to avoid heterocoagulation) in a $100 \times 10\text{ Gm}$ cylindrical column. The material was allowed to settle over a height of 75 cm for time t_1 which depended upon the coarsest particle of the desired size range. The material taken out of the cylinder after allowing it to settle for time t_1 was collected in a bucket fitted with a tap.



The material was allowed to settle again over a height of 30 cm for time ' t_2 ' so as to eliminate submicron (minus 1 micron) size particles. The particles settling at the bottom of the bucket were the desired particles and were collected in another bucket.

The settled particles in the cylinder were wet-ground for 4 hrs. in a ball mill with ceramic balls. The pulp density inside the ball mill was kept around 70-75 pct. The speed of the ball mill was calculated by Fahrenwald's empirical equation²¹.

$$N = \frac{54.2}{(S-s)^{1/2}}$$

N : critical speed

S and s are the radius of ball mill and the

balls respectively, both expressed in feet.

The ground pulp was again allowed to settle and this process was repeated till most of the material was converted into particles of desired size range. The prepared material was washed a number of times and allowed to settle for time ' t_2 ' so as to remove any submicron particle. This also enabled the pH to be lowered to the normal value. Particles of size ranges 1-8 μm and 1-20 μm were prepared by this procedure.

The settling time t_1 or t_2 for different size particles were calculated by using Stokes equation and a tabulated in



Table A, Stokes equation:

$$V_m = \frac{2}{9} \frac{(\Delta - \Delta') r^2 g}{\mu}$$

where

V_m = Terminal velocity of the settling particles

r = Radius of the particle

μ = Viscosity of the medium (Distilled water)

Δ = Density of the particle

Δ' = Density of the medium

$$\text{Settling time} = \frac{h}{V_m} \text{ seconds}$$

h = height of settling in cm (75 or 30 cm)

Δ was taken as 2.425, the density of Illite which is higher than the minerals Kaolinite, Montmorillonite and Hematite. This should ensure minimum proportion of particles coarser than the desired size range. The particle size was always checked under the optical Microscope. The chemical analysis of various size fractions is given in Table B.

Table A : Settling Time for Particle of Different Size Range

Size Range (microns)	Time of Settling
1-8	t_1 = 4 hrs. 11 min. and 24 sec. t_2 = 108 hrs 45 min. and 17 sec.
1-20	t_1 = 40 Mins. and 11 sec. t_2 = 108 Hrs. 45 min. and 17 sec.

(b) Bhilai Ore:

The Bhilai ore was available in 4"-5" lumps. These were crushed first in Blake type jaw crusher and then in Roll crusher. The 0-74 μ m fraction generated during crushing was sieved out for the flocculation experiments as it was thought that they should represent the slime generated in the Plant. About 3.6 pct. of 0-74 μ m slime was generated during the crushing operation. The chemical analysis of this fraction is given in Table B.

Table II : Chemical Analysis of Different Size Fractions

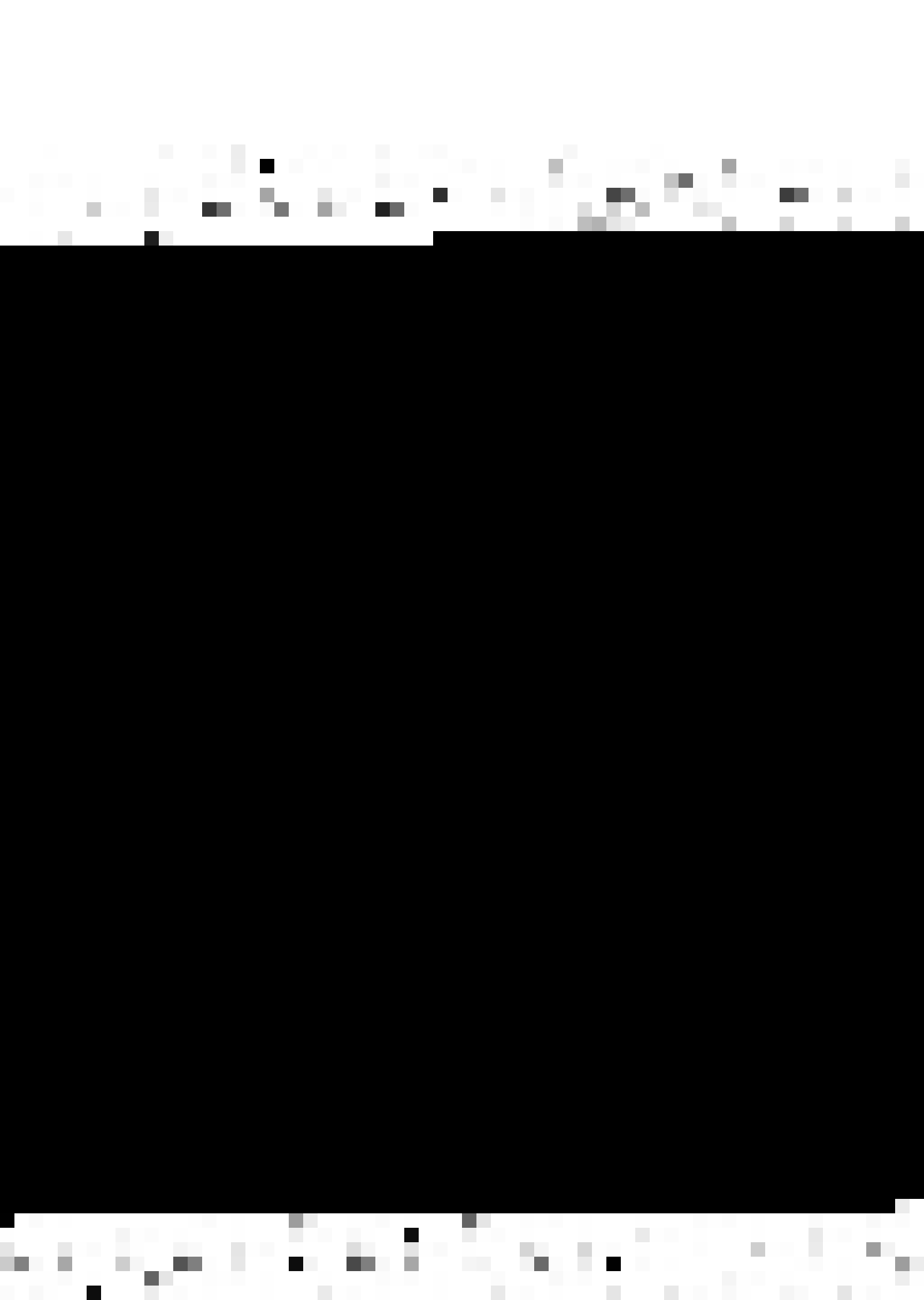
Name of Ore	Lot No.	Size Range (in micron)	Chemical Analysis		
			pct. Fe_2O_3	pct. SiO_2	pct. Al_2O_3
BARBUA [†]	I	0-1	50.4	16.69	21.22
		1-8	65.6	8.84	18.20
		1-20	70.68	10.61	13.94
Bhilai	II	1-8	66.4	10.4	10.00
		0-74	70.4	10.00	10.16
Bhilai		0-74	58.4	16.00	14.88

[†] Chemical Analysis of ore 83-90 pct. Fe_2O_3 , 1.5-2.0 pct. SiO_2
and 4-6 pct. Al_2O_3

2.2 Chemicals:

2.2.1 Flocculant:

- i) Commercial Starch
- ii) Starch Phosphate
- iii) Magnafloc 292, a cationic flocculant supplied by
'Allied Colloids Ltd.', U.K. Method of Solution
Preparation is discussed elsewhere¹⁵.



2.2.2 Dispersant: Sodium Silicate (Na_2SiO_3)

~~This was of H.~~ It contained some insoluble material, so whenever the solution was prepared the insoluble was filtered out.

2.2.3 Modifier: Sodium Fluoride (NaF)

2.2.4 Flotation Reagents:

i) Collector : N-Dodecyle Amine

Hydrochloride: This is a cationic collector, soluble in water.

ii) Frother : Pine Oil.

iii) Commercial Kerosene Oil which was filtered.

2.2.5 pH Controller: NaOH or HCl was used for Controlling pH. Since starch and the mode of preparation of starch solution are very important topics, a separate chapter (Chapter III) is devoted to these.

CHAPTER III

PREPARATION OF STARCH AND STARCH PHOSPHATE
FOR SELECTIVE FLOCCULATION

Starch has been recently considered as a promising selective flocculant for iron ores. Various methods of solubilising starches have been investigated with the aid of a microscope and it has been demonstrated that the rupture of granules is a prerequisite for the effective use of starches²². Mechanical or thermal treatment of the causticised starch decreases the molecule size as inferred from viscosity measurement. The different methods of starch preparation that could be found in literature are stated below.

- (1) Heating with stirring at 90-95°C for 15-20 minutes and cooling to room temperature.
- (2) Heating in autoclave at 130°C for 15 min and cooling to room temperature.
- (3) Heating in an autoclave for 165°C for 15 min and cooling to room temperature.
- (4) Causticising with 1N caustic soda followed by stirring at room temperature for 30 min.
- (5) Homogenising in a blender for 15 min., the solution causticised by method 4.



- (6) Heating at 100°C for 15 min. the solution causticised by method 4.

To rupture the starch granules in order to form a colloidal solution, a weighed quantity of air-dried starch was dispersed in distilled water and solubilised by causticising and/or heating as stated above. To minimise the effects of microbiological decomposition or of chemical degradation, starch solution should be prepared just before use²². (Of course it has been reported that solutions as dilute as 10 mg/litre remained unaltered for 3 weeks or more when properly sterilised by heat²³).

Starch solution prepared by first three methods were not used for flocculation studies by Iwasaki²². The causticising (method 4) and heating at 165°C gave essentially the same microstructure in which the granules were completely destroyed. There was no noticeable difference in the microstructure of the starch prepared by the methods 4, 5 and 6. But the average molecular weight of starch obtained by method no. 5 and 6 was reduced when compared with that obtained by method no. 4. This was indicated by viscosity measurement²².

As the microstructure obtained in two cases (method no. 3 and 4) were similar, it appeared that method no. 4 could be preferred only on the basis of its cheapness or convenience,

otherwise, there appeared no reason why any one of them should be preferred to each other. Both heating and stirring can cause disintegration.

Dicks¹³ reported the following cooking process for making starch solution. The dry starch was put in cold water slurry under agitation and then brought to a boil and held just under boiling (about 95°C) for 15 minutes with agitation. This suspension was then diluted with water to the desired strength and was ready for use. This method which does not involve any use of NaOH is almost similar to the method no. 1.

So far as the 2nd method is concerned, it has been reported by Schoch et al²⁴, that after autoclaving, the solution had somewhat lower intrinsic viscosity than the solution obtained from boiled starch-paste. For this reason, prolonged boiling in the presence of excess pentasol was preferred as a means of solubilising starch prior to fractionation. Use of blender had been suggested to disintegrate swollen starch pastes for fractionation. However, B-fraction (amylopectin) undergoes degradation under violent shearing action seemingly by a mechanical hydrolysis of the molecule. This was indicated by large fall in viscosity²⁴ and also by decrease in settling rate of flocs in flocculation in a flocculation

excellent²². Pregelatinisation in liquid ammonia provided an excellent but somewhat inconvenient method for dissolving granule structure without degradation²⁴. This last method again indicated the importance of OH^- ions and hence of causticising. So it appeared that method (1) and (4) for making starch solution could be used for our purpose. Dick's et al¹³ have reported that the method (1) of making starch solution respond only to the acid modified starch, which has virtually all of the protein removed and has acidic pH. The method does not respond to pearl type of starches. In our case also we could not get a clear solution by this method. However, the method may be successful for phospho-rylated starches.

The starch solution prepared by the first four method have been used as depressant of iron oxide in the soap floatation of activated silica from iron ores²⁵. It has been reported that the solution prepared by method 3) and 4) gave better results (in terms of pct. Fe recovery and pct. Fe in the concentrate) when compared with results obtained while using starch solution prepared by method 1) and 2). Starch solution prepared by method 3) and 4) gave practically same results. So for practical convenience causticising can be adopted for our experiments.

Method 5) of starch preparation may be important as violent shearing reduces the chain length and so may reduce the consumption of starch.

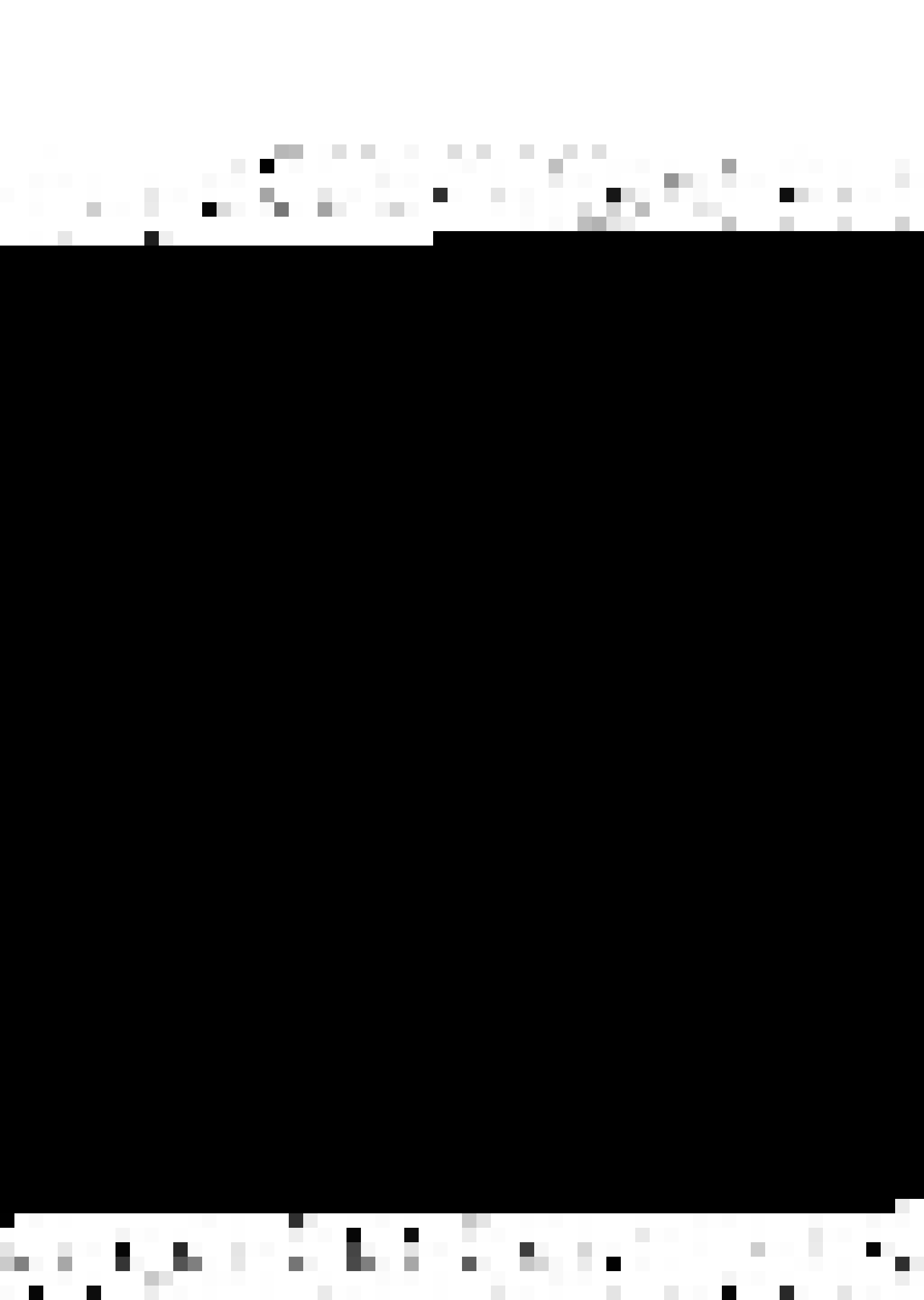
The floatation results obtained with the steam-heated starch solution prepared according to method (6) were not as good as those obtained with the causticised and homogenised starch prepared by method 5). This may be attributed to the difference in the molecular size distribution of the starch in the two cases²².

So on the basis of the above findings following methods can be tried for the preparation of starch solutions.

- i) Heating with stirring at 90°C for 20 minutes and cooling to room temperature. (for Phosphorylated starches only).
- ii) Causticising with caustic soda followed by stirring at room temperature for 30 minutes and
- iii) Homogenising in a blender for 15 minutes and then the solution causticised by method 4.

STARCH PHOSPHATE: Most natural starchy materials contain phosphoryl groups which may be responsible for good flocculating power. Therefore, it was intended to prepare and use starch phosphates. Starch Phosphate was prepared by following methods, the details of which are available elsewhere²⁰.

1. Preparation of Starch phosphate using urea.
2. Preparation of Starch phosphate using H_3PO_4 .



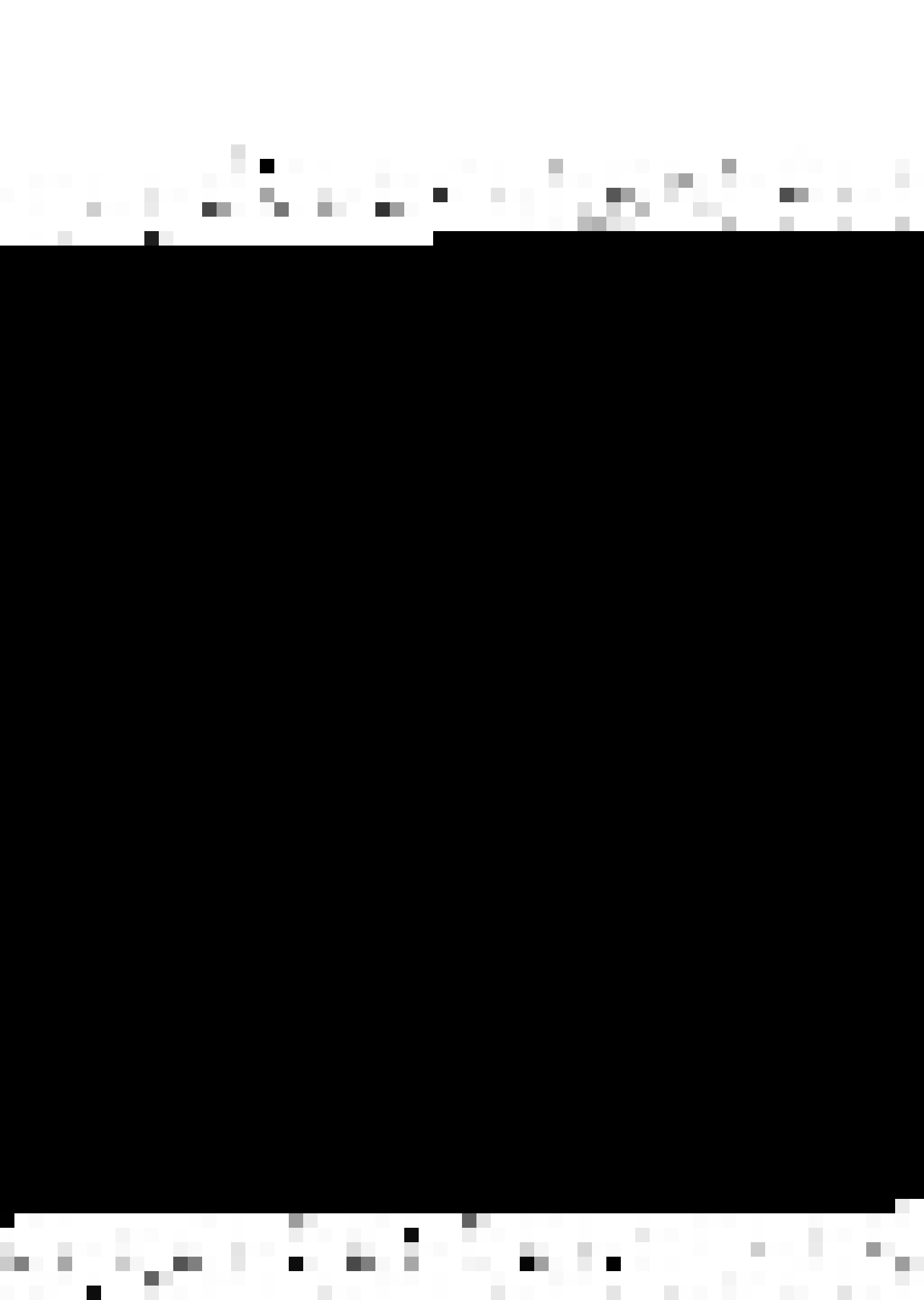
3. Preparation of Starch phosphate using Mono and Misdian hydrogen phosphate.
4. Preparation of starch phosphate using POCl_3 .

The phosphorous content in the starch phosphate prepared by different methods is reported in Table A. Phosphorous content of commercial starch as well as pure starch was also determined and is also reported in Table A.

Table A: Phosphorous Content in Starch Phosphates and other Starches

Name of Starch	Method of Preparation	Phosphorous Content
Starch Phosphate	No. 1	2.38 pct.
	No. 2	0.077 pct. - 1.6 pct.
	No. 3	3.4 pct.
	No. 4	1.4 pct.
Commercial Starch	-	0.012 pct.
Chemically Pure Starch	-	0.014 pct.

Starch Phosphate containing 3.4 pct. P and commercial starch were used for this work.



CHAPTER IV

TECHNIQUES, EXPERIMENTAL AND RESULTS

4.1 TECHNIQUESPreparation of Starch Solution(a) Commercial Starch

(1) Causticising and Homogenising Method (CH): 0.5 gm of commercial starch was taken in a 500 cc beaker and to it was added 3 cc of distilled water. The agglomerates were broken down by a glass rod. Then 3 cc of 0.5(N) NaOH was added the suspension was stirred for 1 minute by the glass rod 0.5 cc of 0.5(N) NaOH was then added when a thick gel formed. The gel was gently mixed for 1 minute and then it was kept still for next six minutes. 1 cc of distilled water was then added and the gel was mixed with the glass rod. Distilled water was slowly added with mild agitation with the glass rod and the volume was increased to 300 cc. This operation was completed in two minutes (total 10 minutes). The diluted was then stirred at 1000 RPM for 15 minutes by a motor stirrer. This was then homogenised in a Balli mixer for five minutes at 16000 RPM. The solution was then transferred to

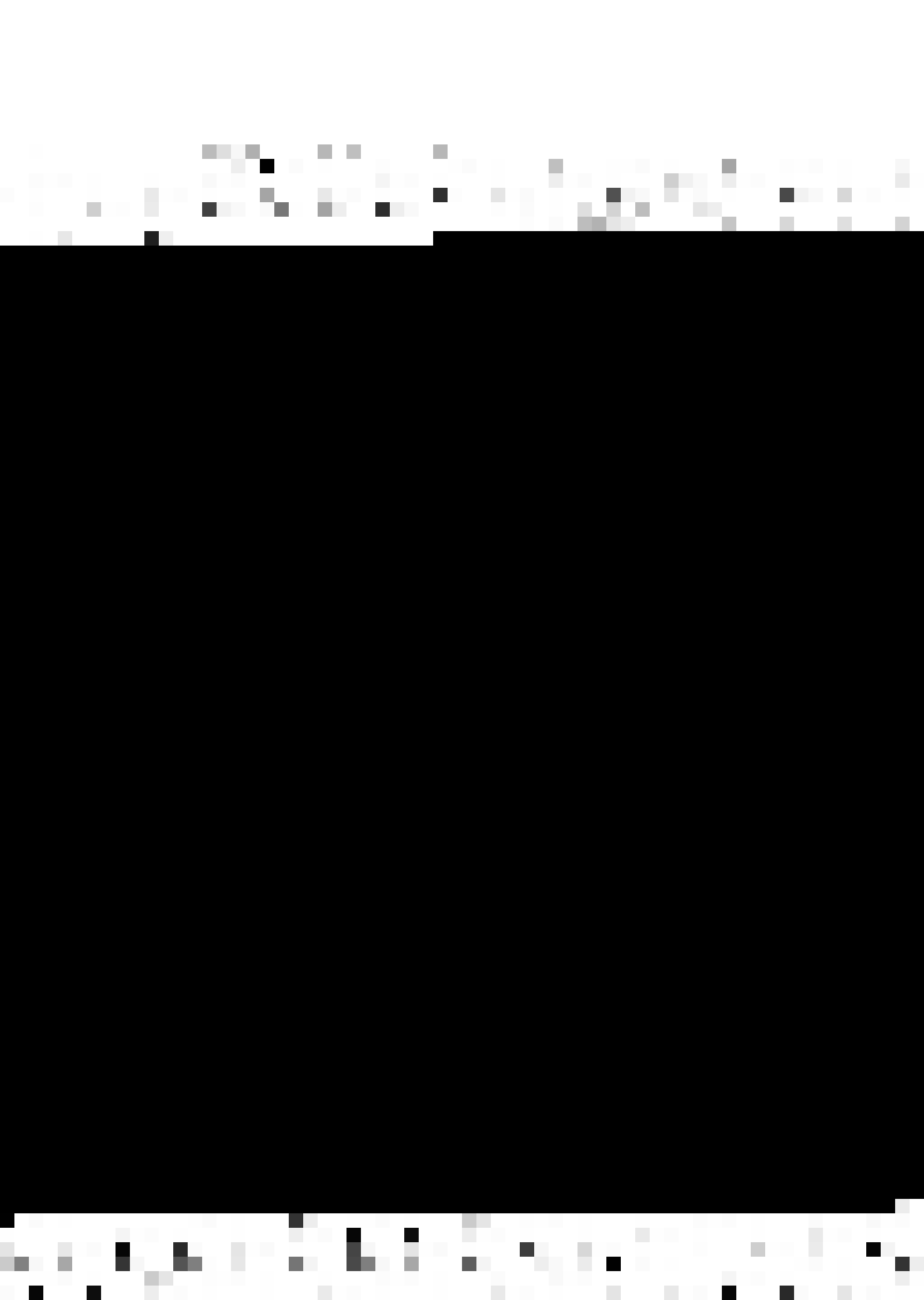


a 500 cc measuring flask and the volume was made upto 500 cc (total time of preparation of starch solution was 30 minutes).

(11) Modified Causticising and Homogenising Method (MCH): This method is essentially similar to the CH method. The only difference is that the stirring step (by motor) in CH method was eliminated. The diluted gel was directly transferred to the mixer and homogenised at 16000 RPM for five minutes. The solution was then transferred to a 500 cc measuring flask and the volume was made upto 500 cc (Total time of preparation 15 minutes).

The starch solution prepared by the above methods were used within 10 minutes of their preparation, during that time it was cooled under tap water. Only in case of ageing variety of the solution it was used after 24 hrs.

(b) STARCH PHOSPHATE: 0.5 gm of starch phosphate was taken in a beaker and to it was added 5 cc of distilled water. The agglomerates were broken down the volume of the suspension was increased to about 3000 cc by adding distilled water and the solution was homogenised in the homogeniser for 10 minutes at approximately 16000 RPM to obtain a clear solution. The rise in temperature was around 65°C. The experiments with starch phosphate had be discontinued later as it was felt



that the results are difficult to reproduce²⁵. The flocs obtained with starch phosphate were very stable. Even intense agitation in a homogeniser couldn't prevent the flocs to reappear and settle after the homogeniser was put off. Similar observation were made with commercial starch, but there was some loss of material after agitation.

Procedure for Selective Flocculation Experiments: The flocculation experiments were performed in a glass apparatus shown in Fig. 1. One litre of distilled water was taken in a flocculating column and to it was added sodium silicate. The stirring was started and after one minute the slime pulp was added. The pH of the suspension was then adjusted by adding 1 (N) NaOH. Seven, minutes after pulp addition, sodium flouride solution was added and after one minute starch solution was added. The pH of the starch solution was adjusted to the pH of the suspension, just before it was added. The stirring was continued for one minute, after the starch solution was added to the system, and then it was gradually reduced to zero in the following minute. The suspension was then allowed to settle for one minute. The unsettled portion was then tapped out from the lower tap followed by the removal of the flocculated/settled portion from the bottom of the apparatus.



The flocculated (settled) portion was treated again by the process just described. This was the second stage of flocculation.

The flocculated (settled) portion obtained at the end of second stage flocculation was again treated by the same process. At every stage in multi-stage flocculation experiments, additional and identical dosages of reagents were used. This was the third stage of flocculation. The flocculated/settled portion obtained after the third stage of flocculation was termed as fraction 'A'.

The three unflocculated/unsettled portions obtained at the end of 3-stages of flocculation were mixed. The mix was then treated in three batches by the same process as stated above. The three flocculated (settled) portions obtained were mixed and termed as fraction 'B'. The unflocculated (unsettled) portions obtained were mixed and were termed as fraction 'C'.

The three fractions 'A', 'B' and 'C' were filtered, dried, weighed and analysed for Fe_2O_3 , SiO_2 and Al_2O_3 .

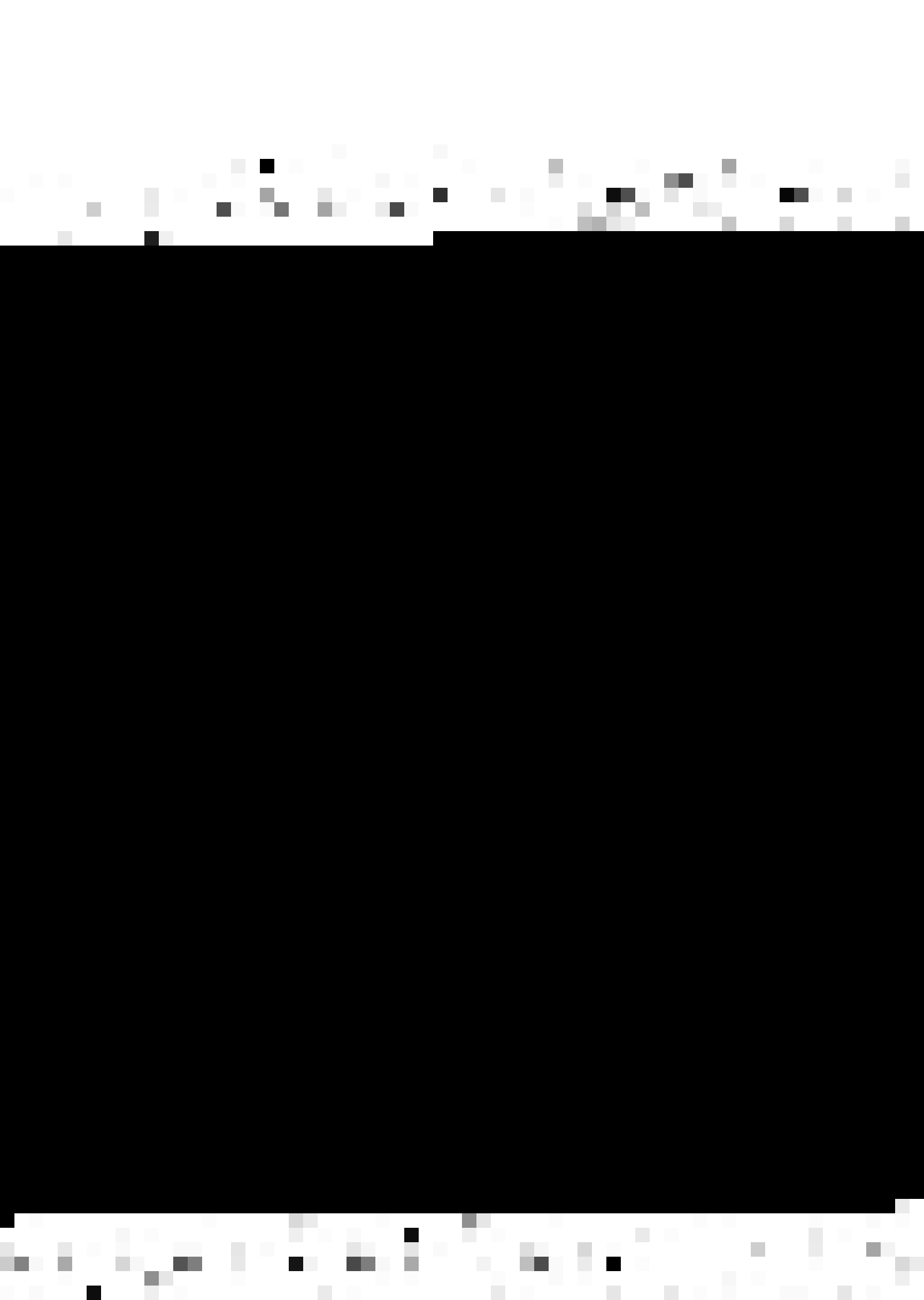
In case of two stage-flocculation experiments the experiment was stopped after the second stage of flocculation. For six-stage flocculation experiments the flocculation procedure, just described was repeated six times. The

unflocculated (unsettled) portion was not treated any further. The concentrate obtained at the end of three stage flocculation was obtained twice and mixed. This was then again treated in 3-stages, thus making the total no. of stages to six.

At every stage some sample was withdrawn from the system for getting it analysed and also to calculate recovery. The total volume of suspension treated in one stage was 1600 cu.

Procedure For Floc-Flotation Experiments: The floc-flotation experiments were performed on (1-8 μ m) Barsua slime and on the synthetic mixture of pure minerals. About one litre of distilled water was taken in a Denver flotation cell and to it was added the pulp (slime/pure minerals). The stirrer of the Denver flotation machine was started at a speed of 1200 RPM. The pH of the suspension was adjusted to 8.5.

After 5 min Magnafloc 292 flocculant was added. The suspension was conditioned with the flocculant for 1 minute. The stirrer was then stopped, so as to allow the flocs to form. After 1 minute the stirrer was re-started and the cationic collector N-Dodecyl amine hydrochloride was added to the system. The stirring was re-started so as to dislodge any mechanically entrained particles (such as of Fe_2O_3 etc.) as well as to condition the system with the collector. After 5 minute of



conditioning with the collector, 2 drops of pine oil were added and after 1 minute the air was allowed to pass through the suspension. The floated material was collected over a period of 2 minutes.

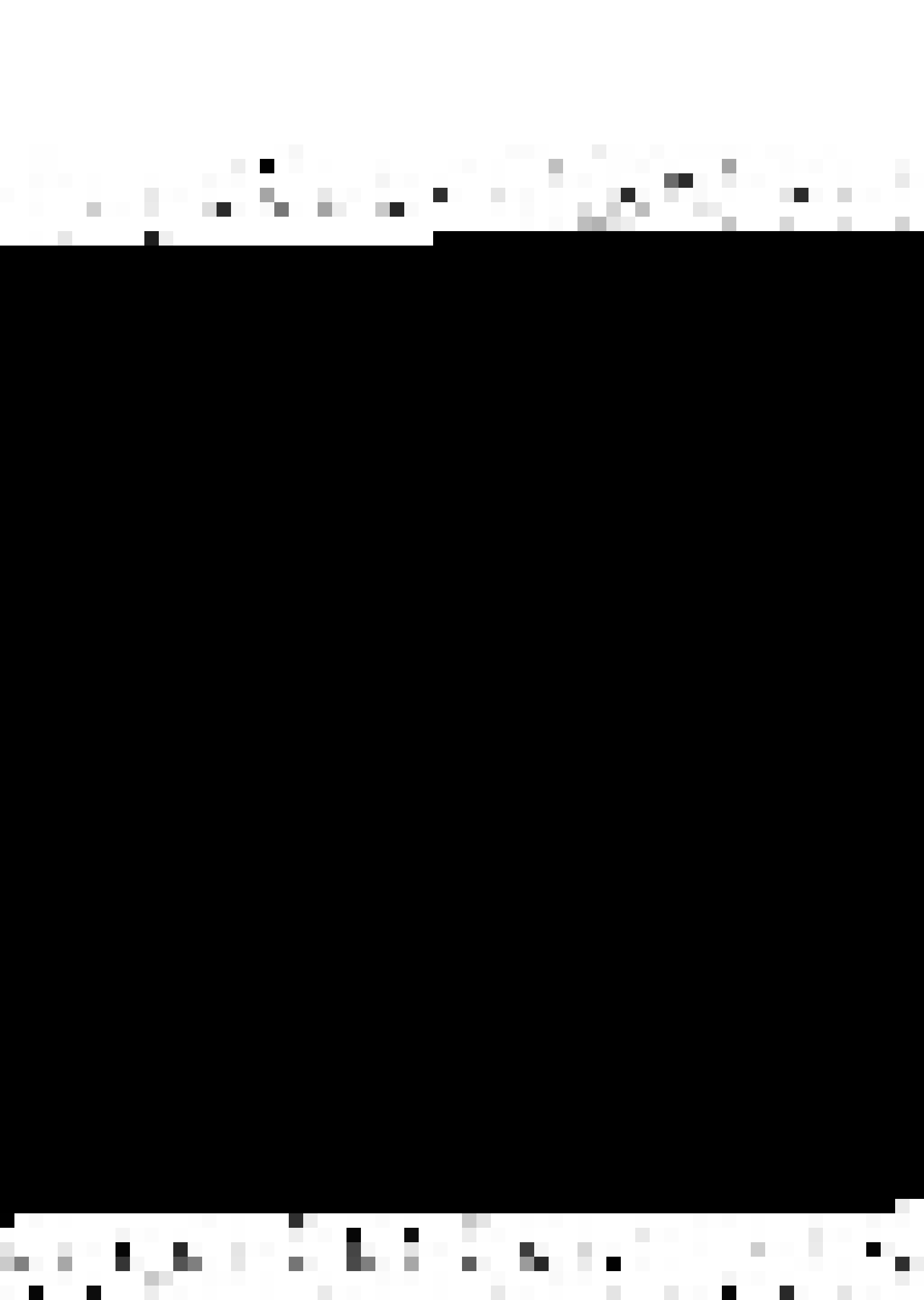
In some experiments kerosene oil droplets were used instead of air to float the material. No pine oil was added in those experiments. The process was similar to the process involving air. After 5 minutes of conditioning with the collector (in case it was used) kerosene oil was added. After 1 minute the stirrer was stopped and the system allowed to separate in two layers in two minutes. The top layer was then removed by adding water at the bottom of the cell. The total volume of the suspension during floc-flotation experiments was 1600 cc and pulp density 1.0 pct.

The chemical analysis of the floated and unfloated fractions were done by dissolving in HCl^{22} (The details of the procedure are reported by Sharma²⁵).

Further details regarding each experiment are provided with the corresponding tables.

4.2 EXPERIMENTS AND RESULTS

Initial experiments were performed with 1-20 μm *S. aurea* slime and with causticised and homogenised (CH) starch. Both ageing and non-ageing variety of starch were tried. The



results tabulated in Table 1, show that although the grade obtained with ageing variety of CM starch is better, the recovery is poor. Sharma²⁵ also showed that recovery of hematite with the ageing variety of CM starch was very poor. So for future experiments it was decided that fresh starch solution (Non-ageing variety) would be used for flocculation experiments.

Some experiments were performed with freshly prepared CM starch, and 1-20 μm particles to find the optimum concentration of starch which would give good selectivity index values. The results are tabulated in Table 2 (Fig. 2). 1-5 ppm starch gave best selectivity indices. But still the alumina percentage is not below 6.0. Similar experiments with starch phosphate gave better selectivity index values. The results are tabulated in Table 3 and plotted in Fig. 3. The best selectivity index value for $\text{Fe}_2\text{O}_3\text{-Al}_2\text{O}_3$ separation ($\text{S.I.}_{\text{FeAl}}$) was 2.09, but still Alumina percentage was not below 7.0 pct.

It was felt that probably liberation is not good at 1-20 μm . Liberation studies referred to in Chapter I also indicate better liberation at 1-8 μm size range. So it was decided to carry out future studies with 1-8 μm size only.

Initial experiments with 1-8 μm particles were performed, under the conditions similar to the conditions under which the



experiments with 1-20 μm were performed. The sodium silicate and sodium fluoride concentrations in both the cases were 100 and 50 ppm respectively and pH was 10.5. No flocs were observed when the system was allowed to settle. The percentage of material collected as fraction 'A' was less than 2 pct. and that collected as fraction 'C' was more than 75 pct starch concentration from 1 to 100 ppm were tried.

Some trial experiments were done in small cylinders (as performed by Sharma²⁵) to see the settling behaviour of the 1-8 μm particles in presence of different concentration of dispersants at pH 10.5. The results tabulated in Table A indicate that probably the particles are self dispersed.

Table A: Effect of Dispersant Concentration on Settling Behaviour of 1-8 μm Particles

Dispersant concentration ppm	pct. settled
0	27.51
25	28.39
50	28.1
75	29.98
150	26.22



Further 3-stage flocculation experiments at 10.5, without any dispersants failed to increase the recovery of fraction 'A' beyond 5 pct. It was thought that apart from high dispersant concentration following factors may be underly poor flocculability of 1-8 μ m particles.

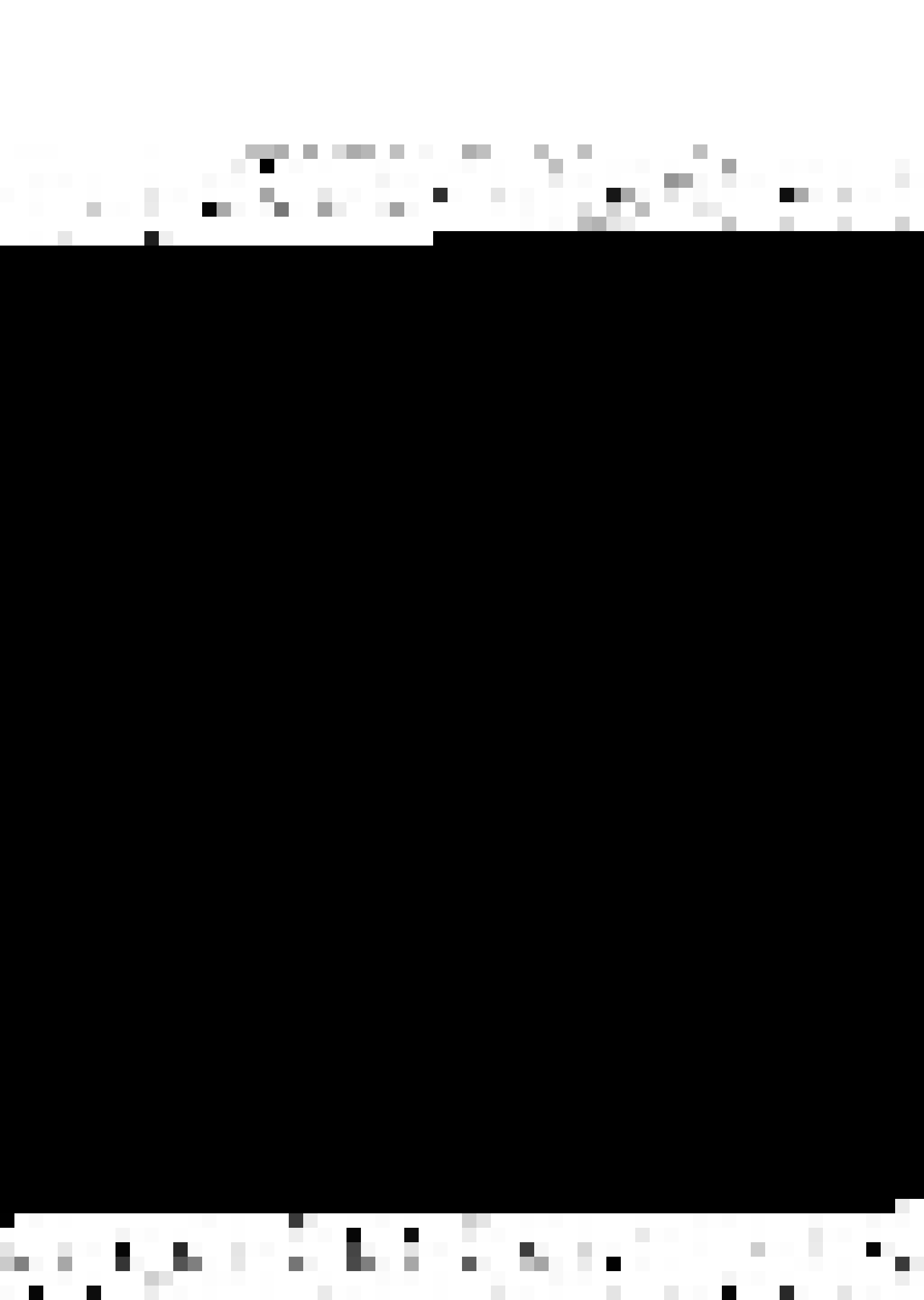
(i) Use of Alkali: Apart from dispersing the clayey possibly it is adversely affecting the flocculation of fine hematite particles also.

(ii) Conditioning Time and Stirring Speed: It was observed that if the unflocculated material was undisturbed for some time (say 3-4 minutes) some fraction of the unflocculated material settled at the bottom of the container. Presumably they were flocs of smaller sizes as 1-8 μ m particles do not settle in such short period. It may be possible that the conditioning time of one minute was not enough for the adsorption of starch molecules on the fine hematite particles (slow kinetics) or the stirring speed was high.

(iii) Particle concentration may be another important factor governing the flocculation of material.

(iv) Clime Coating: It may be possible that fine clay particles are forming a coating over the hematite particles.

(v) Starch Concentration: Since the particles are very fine, it may be possible that the starch concentration used so far was not enough to give good recovery.



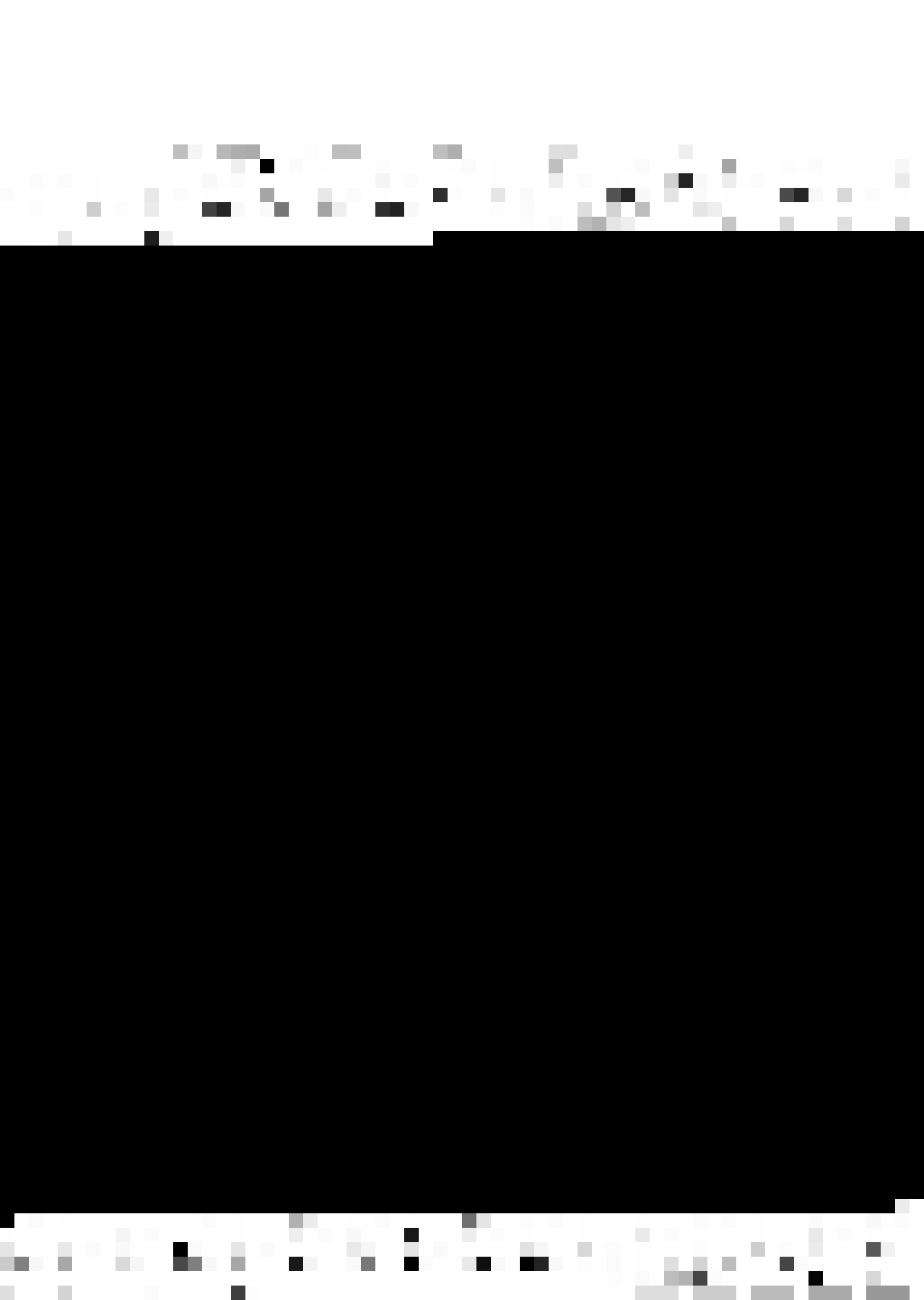
(vi) Method of Preparation of Starch: This factor^{22,26} has been found to be an important.

Few single stage experiments were performed to check the above factors and to find out the conditions, which would enhance flocculation of 1-8 μ m slime particles. The parameters like starch concentration, stirring speed, pH, pulp density, dispersant concentration were studied. The results are tabulated in Table 4. The results indicate that the higher stirring speed reduce the percentage of flocculated (expts. 1 and 2, Table 4) material (expt. No. 1 and 2). Addition of Sodium Silicate, Alkali and higher concentration of starch also reduce the amount of flocculated material (expt. No. 2, 3, 4, 7 and 8). The addition of sodium silicate is probably very critical. Even at lower stirring speed there is only 19 pct. material settling in presence of 100 ppm Sodium Silicate. This percentage of material normally settles at the bottom of the apparatus, whether the flocculant is added or not. Probably the particles are self dispersed and no dispersant should be added. Higher pulp density gave better results in term of percentage material flocculated. The conditioning time and the stirring step in the preparation of starch solution are probably not very important (expt. No. 2, 9 and 10). However since clear solution was obtained even by eliminating the stirring stage and since the results in terms of percentage of material flocculated are similar

the method of starch solution preparation was modified. The modified method did not include the motor stirring stage.

The best conditions for the single stage flocculation of 1-8 μ m Barsua slime fractions where, starch concentration was 200 ppm, pH 6.7, pulp density 4 pct., stirrer speed during conditioning of pulp with starch 1000 RPM and no dispersant was used (Table 4). However, the percentage of material flocculated at 2 pct. pulp density was also quite high. It was felt that high pulp density like 4.0 pct. would increase the entrapment of the unflocculated particles (presumably clayey) inside the hematite flocs. So pulp density of 2.0 pct. was chosen for future experiments with 1-8 μ m particles.

A three stage flocculation experiment was performed, under the conditions stated above and the results are tabulated in Table 5. Surprisingly only 20 pct. of material is coming as fraction A. One possible reason could be that starch concentration is too high. 200 ppm of starch may be good for one stage experiments, but not for 2 or 3 stage experiments since additional dosages (200 ppm) were used in successive stages, resulting in a build-up phenomenon. Some of the starch molecules are adsorbed on the particles. The particles carry them to the subsequent stages of flocculation.



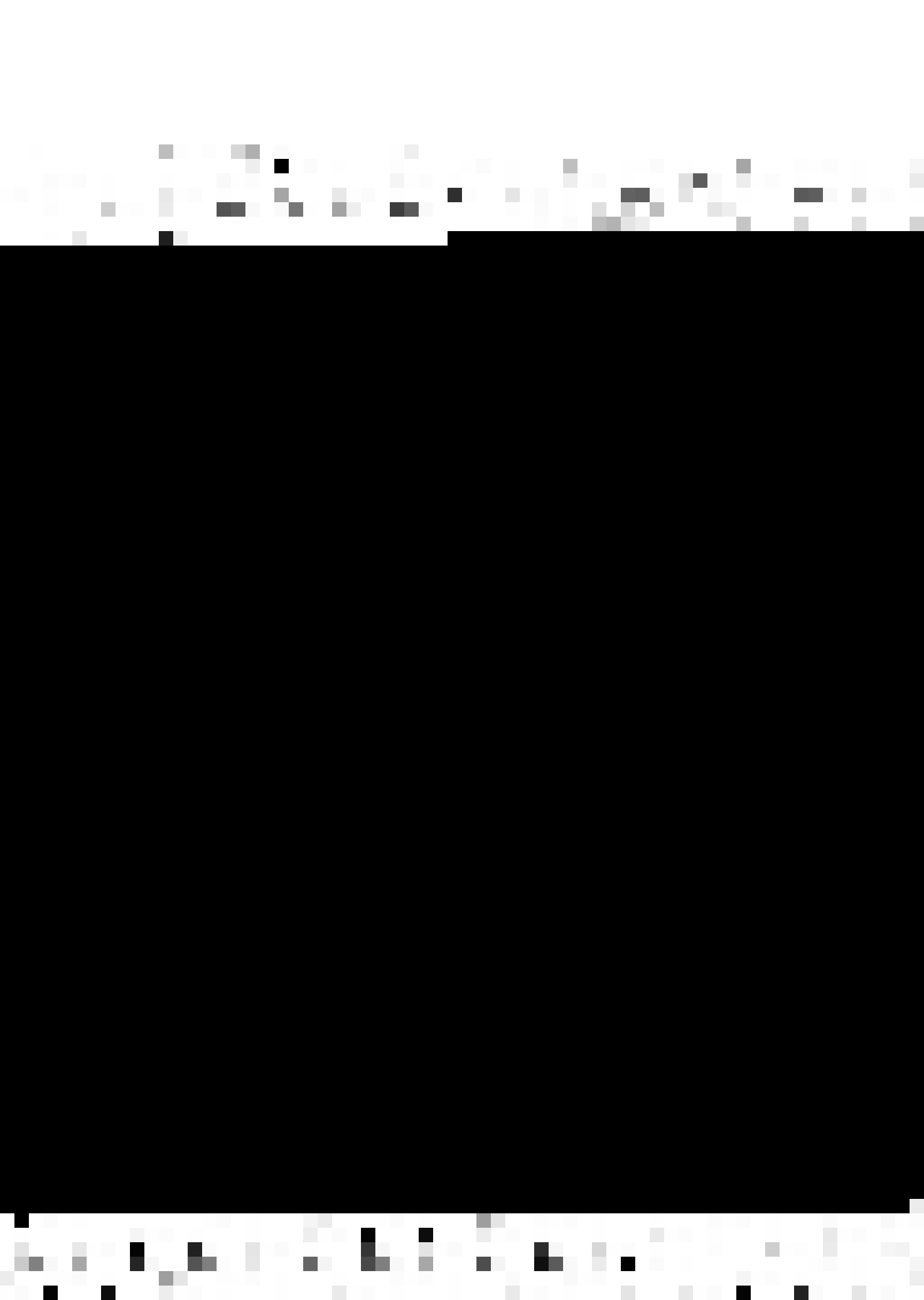
So what may be happening is that the overall concentration during the 2nd stage flocculation is greater than 200 ppm and in 3rd stage flocculation it is still higher. Too high starch concentration during the 3rd stage of flocculation may be responsible for poor recovery. Another possible reason for the poor recovery could be due to inherent mechanical loss on account of successive treatments in multi-stage processes, which might give better grade. So in the next series of experiments the number of stages were reduced to two and various starch concentrations varying from 10 ppm to 400 ppm were investigated. The results are tabulated in Table 6. The results show that recovery falls appreciably when the starch concentration exceeds 10-50 ppm. The recovery figures at the end of two-stage flocculation are high and so it should be possible to carry out three stage flocculation with starch concentrations below 50 ppm. The poor recovery with 200 ppm starch was presumably because it was a high concentration.

So in the next series of experiments on 1-8 μ m Barua slime starch concentration below 50 ppm were used. The pulp density was kept at 2.0 pct and no sodium silicate was used. The starch concentration was varied from 5 to 40 ppm.

Two pH values (7.0 and 10.5) were studied. Although sodium hydroxide has been found to give poor recovery but still it was tried. Since sodium hydroxide is a good dispersant for clays, so the grade of the flocculated portion may be better. In case of experiments at 10.5 pH starch concentrations upto 40 ppm were tried, where as in case of experiments at 7.0 pH starch concentration upto 25 ppm were tried. This was done with the idea to increase the recovery of the flocculated portion.

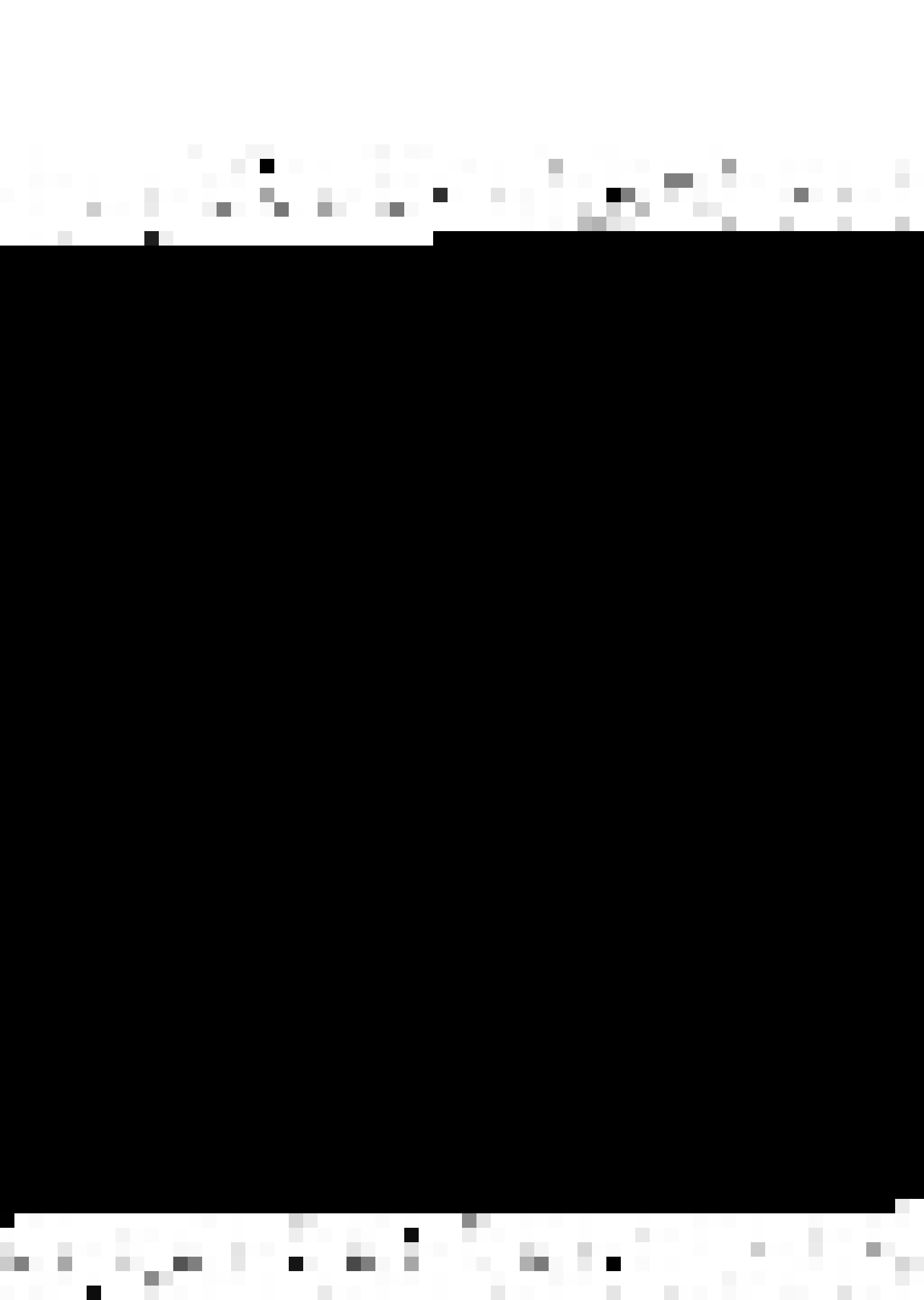
The results are tabulated in Table 7 and 8 and plotted in Fig. 4 and 5 respectively. The data show that grade is better at 10.5 pH but the recovery is poorer. At 7.0 pH the grade is not that good but recovery is better and that is why the selectivity index values are higher. Higher concentration and of starch did not help in increasing the recovery at 10.5 pH.

An investigation was planned to find out whether the above results could be improved by increasing the number of stages of flocculation. Six stage flocculation experiments were tried at 8.5 pH and 10.5 pH. The former was chosen since it is intermediate between the pH values which either gave good grade or poor recovery. Two starch concentrations, 5 ppm and 10 ppm were tried. 10 ppm of starch gave better results



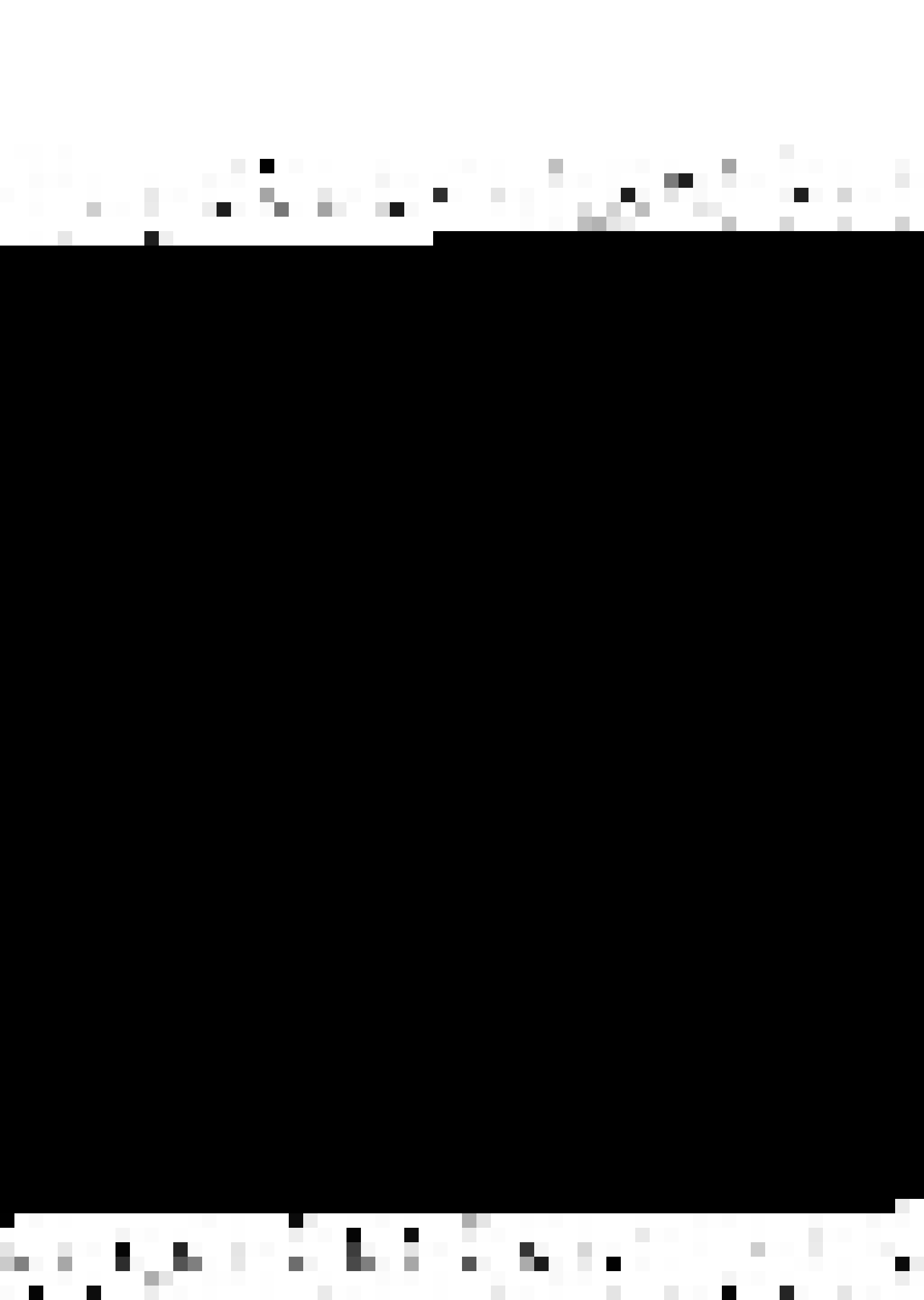
both at 7.0 and 10.5 pH. 5 ppm of starch concentration was chosen because concentration higher than 10 ppm might give poor recovery in the six stage flocculation experiments. The results are tabulated in Table 9-13 and plotted in Fig. 6 and 7. The recovery is higher at 10 ppm starch and at lower pH (8.5). But there is practically no separation even at pH 10.5. This is very surprising as atleast upto 3rd stage similar results are expected as in case of 3-stage flocculation (Table 7 and 8). It may be again mentioned that the samples for flocculation experiments in case of 6-stage flocculation experiments were from a different lot namely lot II. Lot I was received in dry state whereas Lot II was supplied in pulp form. Their mineralogical characteristics could be slightly different.

Next phase of work was done with 0-74 μ m fraction of Barsun Slime and Bhilai ore. This series of experiment was done, so as to see whether the problem with the two are similar or different. Various dispersant concentrations were tried at a fixed concentration of starch. 3-stage flocculation experiments were done. The results are tabulated in Table 13. It seems 15 ppm of dispersant concentration may be chosen as 5 ppm may be too low for the coarse particles. Starch concentration varying from 5 ppm to 20 ppm were tried. In



case of Bhilai ore one dispersion experiment with 100 ppm sodium silicate was done so as to see whether simple dispersion can give good grade. The results are tabulated in Table 14 and 15. The results show that there is some separation but still the selectivity index values are low. Simple dispersion experiment in case of Bhilai ore gave better grade.

It appeared that some other separation process like floc-flotation could be tried. Simple flotation was not tried as the particles are fine and so can not be easily floated. Attempt was made to float the clay particles. It appeared that some other separation technique like floc-flotation could be tried. In floc-flotation experiments the clay particles should be flocculated to make them coarser and then floated. Attempts to float the clayey part were made because of two reasons. Firstly the clayey minerals are lighter than hematite so it should be easier to float them. And secondly the collector consumption should be lower since the proportion of clayey is smaller. The ZPC (Zero Point of charge) of clay and hematite is less than 2 and 7 to 8 respectively. An intermediate pH like 5-6 should be chosen for cationic flotation experiments. This pH should also enable to flocculate clayey particles by cationic flocculant. Experiments below pH 7.0 resulted in settling of all particles



probably because of the combined effect of heterocoagulation and flocculation. These experiments were done in 50 cc test tubes. Similar trial experiments at pH 8.5 and 10.5 showed that the flocculation was not that severe at these pH values. For floc-flotation experiments pH 8.5 was chosen, as too high pH would enhance the adsorption of cationic collector on hematite particles.

Floc-flotation experiments were carried out at a fixed flocculant concentration and varying collector concentration. The main idea of adding the flocculant was to flocculate the clayey part. For this, the flocculant concentration had to be fixed. Various flocculant concentrations were tried. The experiments were performed in small cylinders and the results are tabulated in Table 16. Flocculant concentration of 1 ppm which corresponds to approximately 50 pct. material settled was chosen for floc-flotation experiments⁹.

⁹ This was because 30 pct. of the total material taken in the cylinder would always be below the nozzle through which the unsettled portion is taken out. So actual material that settles is only from the top 70 pct. of the material taken in the cylinder. The slime contains around 30 pct. clay mineral. So 30 pct. of the 70 pct. material at the top and the 30 pct. of the material already in the portion below the nozzle, make total of approximately 50 pct. of material settling at the bottom of the cylinder.



The results of flocc-flotation experiments on 1-8 μ m Barsua Slime and Synthetic mixture of pure mineral are tabulated in Table 18 and 19 respectively. The recovery of unfloated material decreases with increasing collector concentration. Although there is not much separation in case of Barsual slime there is an optimum concentration of collector which gives good grade. The data are plotted in Fig. 8.

In case of experiments with kerosene oil better selectivity is obtained without any collector. It may be possible that kerosene makes the clayey particles hydrophobic.

The results and observations are summarised in Appendix Table III, page 37(a) to 37(c).

APPENDIX TABLE III

Fig. Table No.	Experiments and Materials	Observations
1	Flocculation Apparatus	
1	Flocculation of 1-20 μ m Barsua slime lot I with aged and non-aged starch (CH)	Aged starch gives better grade (39%) but poorer recovery (3.42) S.I. Fe, Al 1.
2	Flocculation of 1-20 μ m (non-aged) Barsua slime with commercial starch	Best selectivity indices for 1-5 ppm starch (S.I. Fe, Al 3.1, S.I. Fe, Al 1.6)
3	Experiment with phosphorylated starch	Better selectivity than in the case of starch; best S.I. for 15 ppm (2.3) %.

EXPERIMENTS ON 1-8 μ m BARSUA SLIME LOT I

4	Experiments with different pH, pulp density, stirring speed and dispersant concentration	Better recovery with higher pulp density, lower pH, lower speed for stirring and no dispersant.
5	Three stage flocculation for 200 ppm starch	Low recovery probably due to high starch concentration
6	Means grade in pct. Fe_2O_3	Means recovery of Fe_2O_3 1: 3.0.
	S.I. means Selectivity Index.	
7	From this value onwards, S.I. stands for Al Fe, Al.	



Fig. Table
No. No.

Observations

- 6 Two stage flocculation

lower recovery when starch concentration exceeds 10-50 ppm.

4 7 3-stage flocculation at 7.5 pH

There is some separation, but not much change in grade with starch concentration. Max. recovery at 20 ppm (G 87)
Higher c.i. values for 1-8 µm (2.66) than for 1-20 µm

5 8 3-stage flocculation at 10.5 pH

Better grade than at 7.5 pH (G 83) but poorer R 21, c.i. 1.53

Best selectivity index at 5 ppm starch 1.6.

EXPERIMENTS ON BARBOSA SLIME LOT II 1-6 µm6 9 and 6-stage flocculation at 8.5 pH
10 for two concentrations of starch

Recovery decreases with increasing no. of stage but no significant change in grade. Almost no separation takes place.

7 11 and 6-stage flocculation at 10.5 pH for two
12 starch concentration.

" " " "

- 13 Effect of Sodium silicate concentration on the recovery of 1-74 µm
Bhilai ore (Three stage flocculation)

Recovery decreases with increasing sodium silicate concentration.



Fig. Table No.	Experiments and Materials	Observations
- 14	3-stage flocculation of 0-74 μ m Bhilai ore	There is some separation but still the selectivity index values are very low. Max. recovery (1.59.2) at 10 ppm starch. Better grade at higher dispersant concentration.
- 15	3-stage flocculation of 0-74 μ m Barsua slime	There is some separation.
- 16	Flocculation of 1-8 μ m Barsua slime by cationic flocculant (Test in small cylinders)	Flocculant concentration of 1 ppm may be used for floc-flotation experiments.
- 17	Floc-flotation of 1-8 μ m Barsua Slime	Little separation. Surprisingly more float in float.
8 18	Floc-flotation of Synthetic mixture (1-8 μ m) of pure minerals	There is some separation. Best selectivity index value obtained is 1.75 at 10^{-5} m/l collector concentration.
- 19	Floc-Flotation of Synthetic mixture (1-8 μ m) using Kerosene droplets instead of air.	High selectivity without any collector (1.55) 0.67, 0.85 in the non-float. Float assayed 37 pct. Fe ₂ O ₃ .

CHAPTER V

SUMMARY AND CONCLUSION

Some flocculation studies have been made by Natarajan et al³⁹ on Indian hematitic ore fines. They have reported amount of starch, amount of CaCl_2 and pH to be significant variables. However, their work on Bhadravati ore assaying 55.7 pct. Fe, 3.2 pct. SiO_2 and 4.2 pct. Al_2O_3 is of little use to us since no selectivity study was made. Our work is probably the first attempt regarding selective flocculation of Indian iron ore/slime.

1. The phenomenon of differential grinding is clearly indicated through different analysis of sized fraction (vide Summary Table II Chapter I). A cut at 15 μm provides best S.I._{Fe, Si} equalling 1.3 whereas the best Fe-Al separation is possible at 8 μm (S.I. 1.72). Larger proportions of Al and Si come in the finer fractions - more Al than Si. This is probably due to softness of clay particles compared to quartz and hematite.

Summary Table I data based on densitometric fractionation indicate better liberation between Fe-Si than Fe-Al upto 15 μm . Liberation becomes appreciable below 15 and 8 μm but even then it is far from complete. (S.I._{Fe, Al} are 1.36 and 2.02 respectively).



2. Selectivity noticed in Barsua Washing Plant operation is due to the combined effects of the above-mentioned factors: differential grinding and small extent^{of} liberation. A typical washing test⁴⁰ on soft laminated and lateritic ore performed on 23.10.1976 gave the following results: 700 T/hr. feed assaying 57.8 pct. Fe, 2.8 pct. SiO_2 and 7.2 pct. Al_2O_3 gave 240 T/hr + 10 mm lump assaying 60.9 Fe, 1.9 SiO_2 , 4.9 Al_2O_3 , 260 T/hr - 10 + 2 mm material assaying 59.9 Fe, 2.3 SiO_2 , 5.1 Al_2O_3 and 172 T/hr slime assaying 42.8 Fe 12.7 SiO_2 and 20.3 Al_2O_3 . S.I._{Fe,Al} for + 10 mm and - 2mm. Slime is computed as $(\frac{60.9}{4.9} \times \frac{20.3}{42.8})^{\frac{1}{2}}$ or 2.4. Somewhat different analysis for the slime provided by R.C. Mohanty (53.5 pct. Fe, 9.2 pct. Al_2O_3) gave re-computed S.I. as 1.45. For 10 mm cut and 2 mm cut, S.I. values are 1.35 and 1.26 respectively. Another set of verbally communicated data (normal washing: washed fraction-Fe 60.0 Al_2O_3 4.0, slime - Fe 48.0 Al_2O_3 9.0, polymer washing: washed fraction - Fe 60.0 Al_2O_3 2.0, slime - Fe 46.0 Al_2O_3 12.0) gave S.I._{Fe,Al} as 1.7 for normal washing and 2.7 for polymer washing.

Thus it is clear that selectivity index values in the plant are limited^{within} 1.5-3.0 due to partial liberation and differential grinding. Polymer washing corresponds to selective dispersion of clay particles and provide only slightly

better S.I.

3. Figs. 2 and 3 (Tables 2 and 3) show that for 1-20 μm Barsua slime particles some selectivity is obtained with lower concentrations of starch and starch phosphate.

4. While experiments with finer particles 1-8 μm were necessary in view of better liberation, difficulties were experienced in terms of flocculability in presence of dispersants. Sharma noticed²⁵ similar difficulty with finer particles such as 0-2 μm pure Fe_2O_3 particles.

5. Starch concentrations exceeding 10-50 ppm produce poor flocculability. It seems that at high concentrations a complete monolayer of adsorption renders a particle non-amenable for contact with another. 50 pct. monolayer adsorption is optimal for inter-particle bridging and flocculation^{27-29,30}.

6. Usually iron-silicon separation has been found to be better than iron-aluminium separation, as adjudged through S.I. values. This was probably due to better liberation of quartz and easier separation of iron oxide through flocculation vis-a-vis quartz^{9,11} rather than clay²⁵.

7. Fig. 4 Table 7 indicate that higher S.I. is obtained for 1-8 μm (2.66) than for 1-20 μm particles. This is



distinctly a liberation effect and better results are difficult to obtain without improved liberation (vide Summary Table I).

8. Higher pH and dispersant concentration produce better grade but poorer recovery. Repeated flocculation lowers recovery without much improvement in grade. Somasundaran et al^{9,10} as well as Dicks et al¹³ have noted general reciprocal relationship between grade and flocculation recovery.

9. Sharma's experiments²⁵ with fully liberated pure mineral particles show that hematite-clay separation is not ideally selective with starch as flocculant. With 50:50 mixture of 1-8 μ m hematite-clay particles treated as 1 pct. suspension with 40 ppm starch, 100 ppm sodium silicate and 50 ppm sodium fluoride, S.I. for kaolinite, Montmorillonite and Illite as clay have been only 3.58, 3.12 and 3.36 respectively. Grade and recovery values were near 76-78 p.c. Much better S.I. values are desirable. Inferior results with Barsua slime (S.I. 2.66) are attributable to incomplete liberation and problem with fine particles.

10. Experiments on 0-74 μ m Barsua slime and Bhilai ore did not give any good selectivity.

11. Recently, there has been a strong research interest in floc-flotation. It has been recommended³⁵ that mineral slimes may be adequately flocculated and then extra flocculant may

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improve the charge conditions on the floc to make it more floatable. Other references on floc-flotation of clay minerals^{36,37} and quartz and sulphide slimes³⁸ are available.

Floc-flotation experiments with mechanical mixtures of hematite-clay pure minerals gave S.I. 1.95 using cationic flocculant and collector and air bubbles for flotation. ~~When and collector and air bubbles for flotation.~~ When Kerosene droplets replaced air bubbles, no collector was necessary. 85 pct. recovery of hematite and 67 pct. assay in the sink coupled with only 37 pct. Fe_2O_3 in the float accounted for S.I. equalling 1.85. Since the separation involved efficiency and selectivity of two steps instead of one, S.I. values were inferior to those obtained for flocculation of mixtures of pure minerals. 1-8 μm iron ore slimes gave inferior results due to liberation problem.

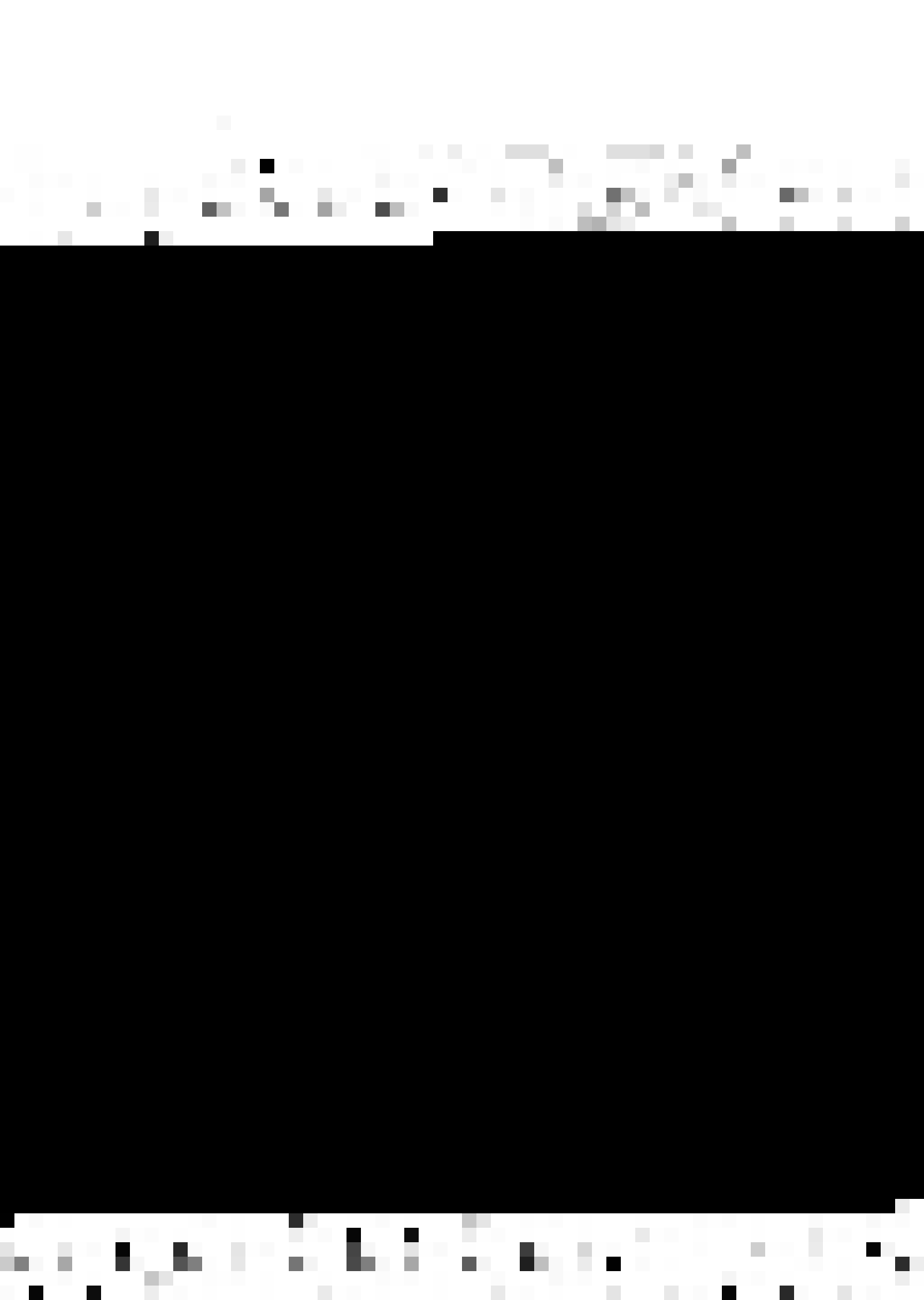
Reasons for limited success in Selective Flocculation

12. Selective flocculation and floc-flotation have been reported to be industrially successful in Tilden Mine operations¹⁸ and for Mesabi range ores¹³ and non-magnetic taconite ores¹². But in such cases, quartz is the principal gangue material and nearly complete liberation have been reported for 25 μm particles. As a matter of fact, coarser and liberated quartz particles were there which could be easily removed by cationic flotation.

13. In the case of Barsua slime, liberation is much more difficult and possible ~~only~~ slightly below 8 μm and appreciably only below 2 μm . That hematite is difficult to flocculate at such small sizes has been indicated by Sharma²⁵ as well as Colombo³¹ for semi-industrial operations.

14. Hematite slimes can be flocculated if dispersants are avoided and starch dosage is increased. The former results in poor dispersibility of clay minerals and later in poorer selectivity. Somasundaran et al¹⁰ and Friend et al³² have reported that selectivity for hematite-quartz separation is high at low starch concentrations. If starch concentration is increased, even quartz gets flocculated lowering selectivity. Re-flocculation gives better grade because starch is not strongly adsorbed on quartz^{9,32}. These considerations introduce an inherent dilemma ^{as} regards usage of larger quantities of dispersant and flocculant.

15. Poor selectivity for quartz and silicate minerals may be caused by hard water or presence of Ca^{++} and Mg^{++} . Ca^{++} ion is known as an activator for adsorption of starch on quartz²². Clarke and Cooke observed⁴² high adsorption of Ca^{++} and Mg^{++} on quartz, at high pH above 10-10.5 as CaOH^+ and $\text{Mg}(\text{OH})_2$. Iwasaki et al⁴³ reported flocculability of quartz at high pH in presence of Ca^{++} and Mg^{++} unless sequestering agents like silicate or polyphosphates are used. Flocculation of Kaolinite by polyacrylamides has been reported to be augmented by Ca^{++} ion³³.



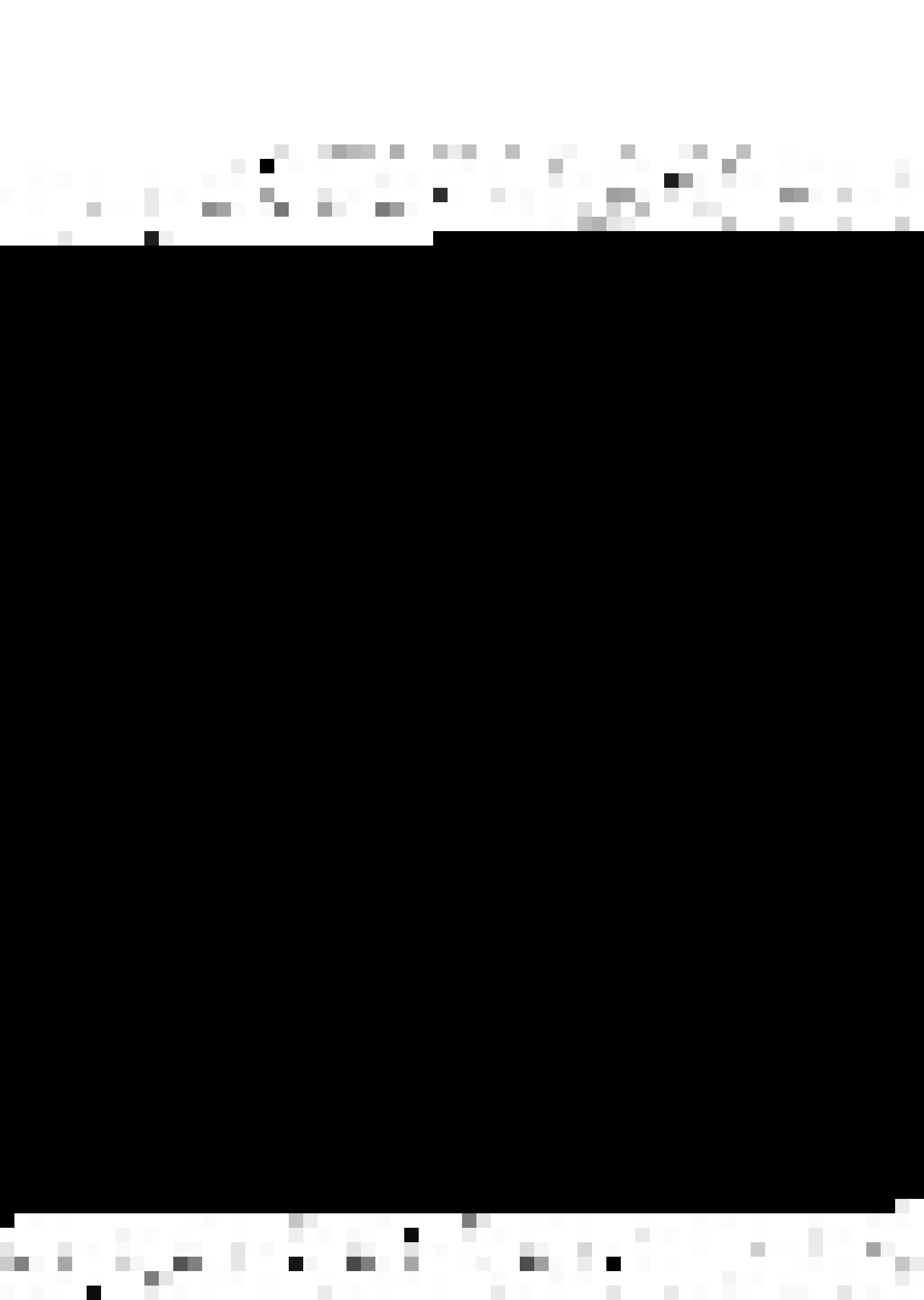
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In our experiments, even though distilled water was used, the filtrate from the pulp in flocculation experiments contained around 80 ppm Ca and 12 ppm Mg as ions (No iron was detectable). It is conceivable that these ions originating from clay minerals³⁴ may self-activate the said minerals with reference to starch adsorption resulting in poor hematite-clay separation which may not be significantly improved by repeated flocculation.

Variables pertaining to the use of Starch

16. While it is known that starch is the most versatile flocculant for hematite, better than polystyrene sulfonate⁹, the details regarding the starch molecules and their constituents, as they are obtained from diverse sources, are extremely complex meriting separate discussion.

17. The early works^{23,44} on starch as depressant or flocculant for hematite show that while all or most starches are adsorbed on hematite, there are appreciable differences on adsorbability, flocculability depending upon the source and method of preparation. Major constituents of starch are amylose (lower mol. wt. $\sim 10^5$, straight chained) and amylopectin (higher mol. wt. $\sim 10^6$, long and branch-chained with more phosphoryl group)²⁶ and there may be transitional molecules



of intermediate size, chain length, branching and number of reactive groups²⁴. It has been shown that most suitable starches for flocculation are anionic^{14,45} and high in amylopectin^{22,46}. Iwasaki and Lai reported²² that amylopectin is more adsorbable than amylose on hematite and quartz. This has been contradicted by Schulz and Cooke²³ who have reported higher adsorbability of amylose. Some work in our laboratory⁴⁷ has clearly established superior adsorbability of amylose on hematite on a molar basis. The results obtained by Iwasaki et al²² were reported on mg/m² basis and can be easily explained in terms of much higher molecular weight of amylopectin. However, in spite of lower adsorbability, amylopectin may be more crucial than amylose for mineral flocculation. Whether this is due to greater chain length or branching or phosphoryl group content is not precisely known.

Chang⁴⁸ has reported oxidised starch to be ^{the} best in selectively adsorbing on hematite particles. Natarajan et al³⁹ has reported that Anilox AF, an oxidised starch has superior flocculating power than Anilox AT which is also oxidised starch but probably of lower molecular weight. Iwasaki and Lai²² have emphasized that rupture of starch granules is an important prerequisite. Causticising/homogenising not only solubilises but also lowers the molecular weight of starch by degradation and makes it 'more effective, weight-for-weight,



in closing the surface' of hematite. This however may or may not correspond to different adsorbability on a strict molar basis.

It is evident from the above discussion that there are many vital factors regarding the use of starch in a flocculation system some of which may be listed as (i) amylose and amylopectin content (ii) molecular size of the two as well ^{as of the} intermediate constituents (iii) branching in the above constituents (iv) number of reactive groups such as phosphoryl group etc in the above and their respective natures in terms of charge density and reactivity in the bulk as well as on the surface phases (v) effect of treatment such as oxidation and of the method of dissolution on factors (ii) to (iv) and lastly (vi) the influence of electrolytes such as dispersants, pH regulators, sequestering agents, natural constituents in hard water, ions liberated from minerals etc. on the physico-chemical behaviour of starch constituents.

In this investigation, the above factors were not deeply studied for two reasons: firstly, most starches including our samples demonstrate sufficient flocculating power in appropriate concentrations; secondly, no detailed work has been done regarding the role of the above-mentioned factors on selectivity of mineral pair systems. A strongly flocculating reagent does not necessarily provide better selectivity.



(In this connection it may be mentioned that polyacrylamide is not as selective as starch and yet it produces larger open structural flocs from which mechanically occluded impurities can be more easily elutriated⁸). Nevertheless, varieties of starch should be used for future comparative studies after the starch samples have been well-characterised and one is in a position to evaluate the individual contributions of the above factors such (i) to (vi).

18. The possible reasons (so far identified by us) for poor selectivity in our flocculation results are (a) poor liberation (b) difficulty in flocculating and selectively flocculating very fine particles (c) the role of Ca^{++} , Mg^{++} (d) lack of knowledge regarding the right kind of starch or for that matter the best possible flocculant to be chosen. One more reason may be suggested: the inherent difficulty with clay systems. Clay particles are known to have negative charges on the main cleavage plane as well as positive charges on the lateral edges. This enables clay particles to hetero-coagulate even on negatively charged mineral particles and provide slimy coatings⁴⁹. This of course can be off-set by judicious use of anionic dispersants and sequestering agents. Nevertheless, poorer selectivity for clay-hematite systems²⁵ compared to quartz-hematite^{9,10} system is evident.



Final Comments

19. While we have not achieved the desirable separation in the case of Barsua slime, the nature of the difficulties associated with such problems has been understood. A clearer picture regarding the mineralogical complexity has emerged. Better success may be achieved with other Indian iron ores corresponding to better liberation at equivalent sizes and containing lesser proportions of clayey materials in the gangue e.g. Kudremukh deposit which is hematitic and magnetitic.

Further studies should be directed towards (1) Mineralogical composition of other Indian iron ores using X-ray, Scanning E.M. techniques etc. as done by us (2) selective flocculation studies on pure mineral systems such as hematite-clay etc. for the discovery of optimal physico-chemical conditions, which may result in S.I. much larger than 3-4.

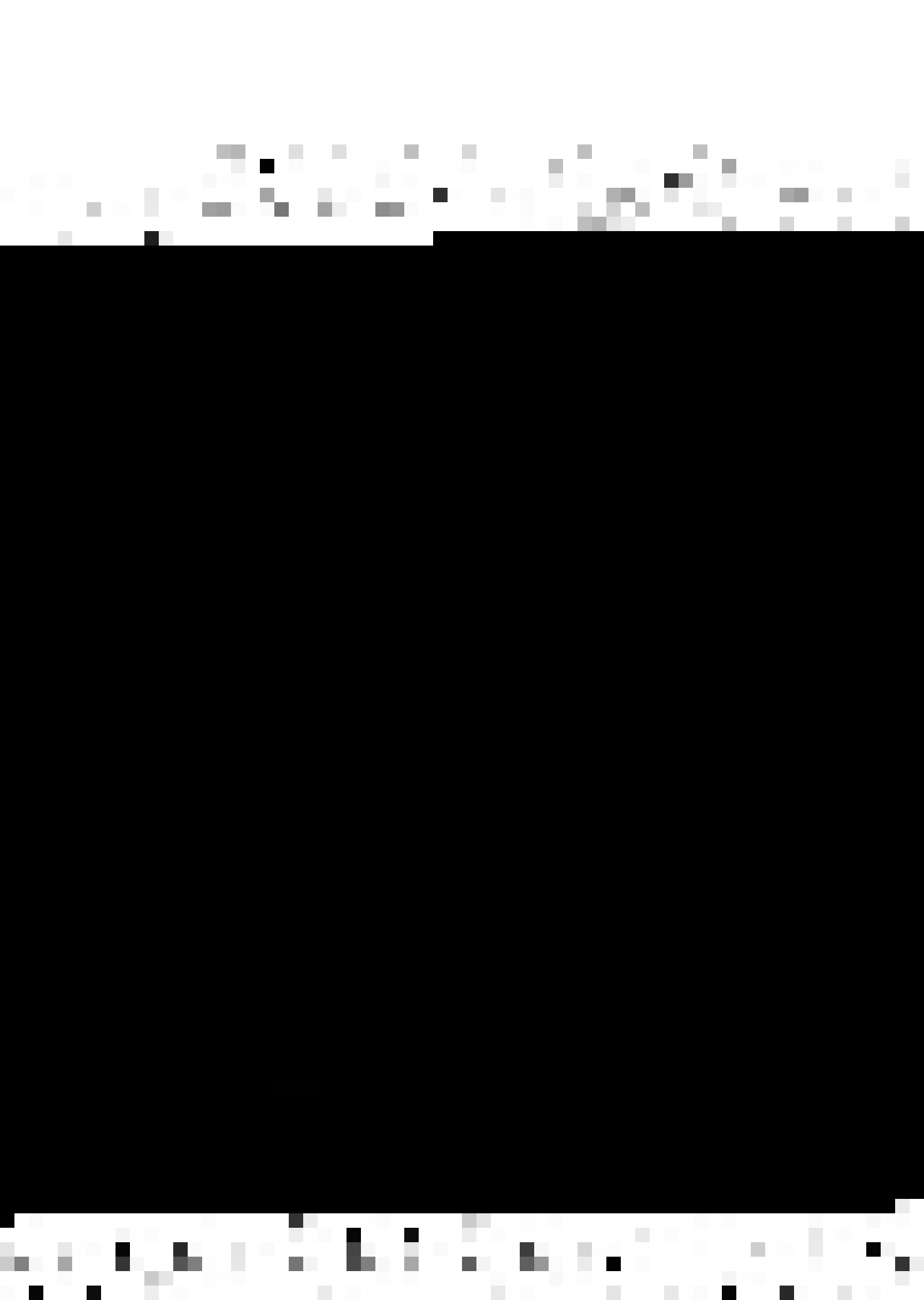
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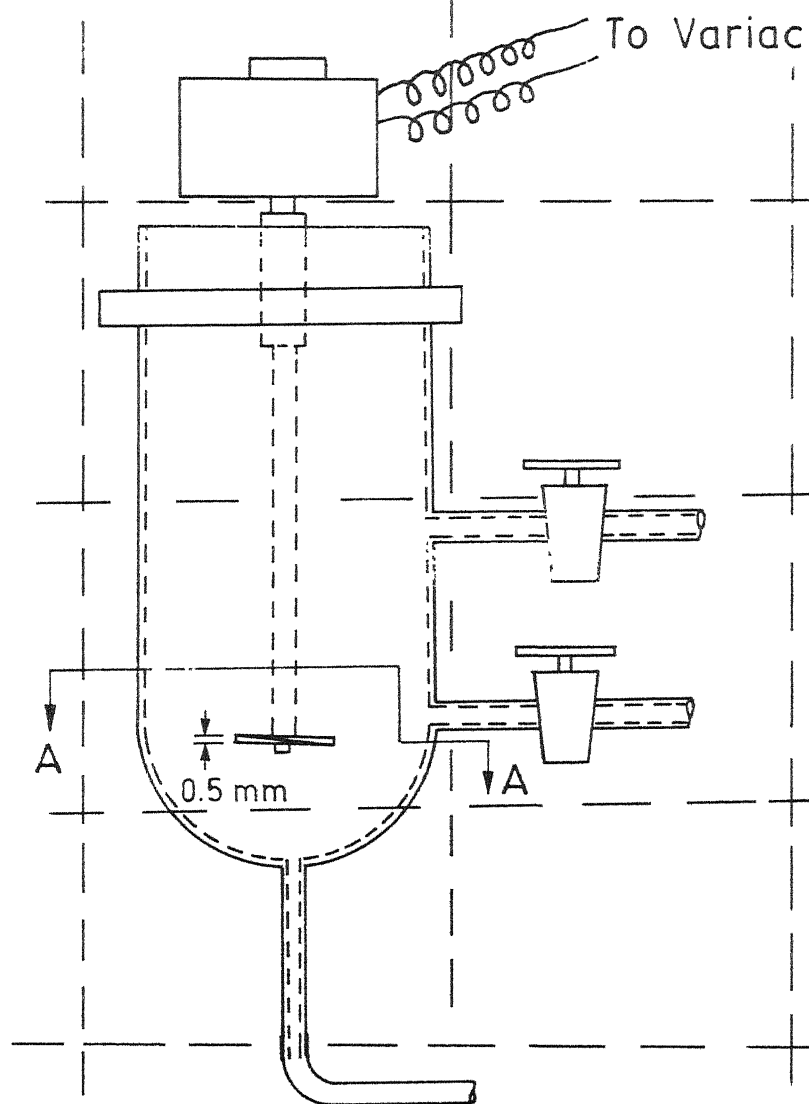
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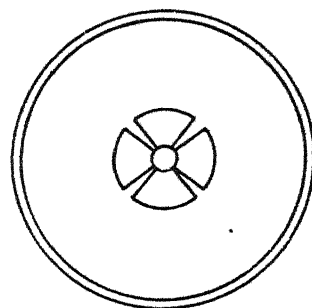


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Scale 1:3



Section on A A

Fig. 1 - Flocculating column.
(Schematic diagram)



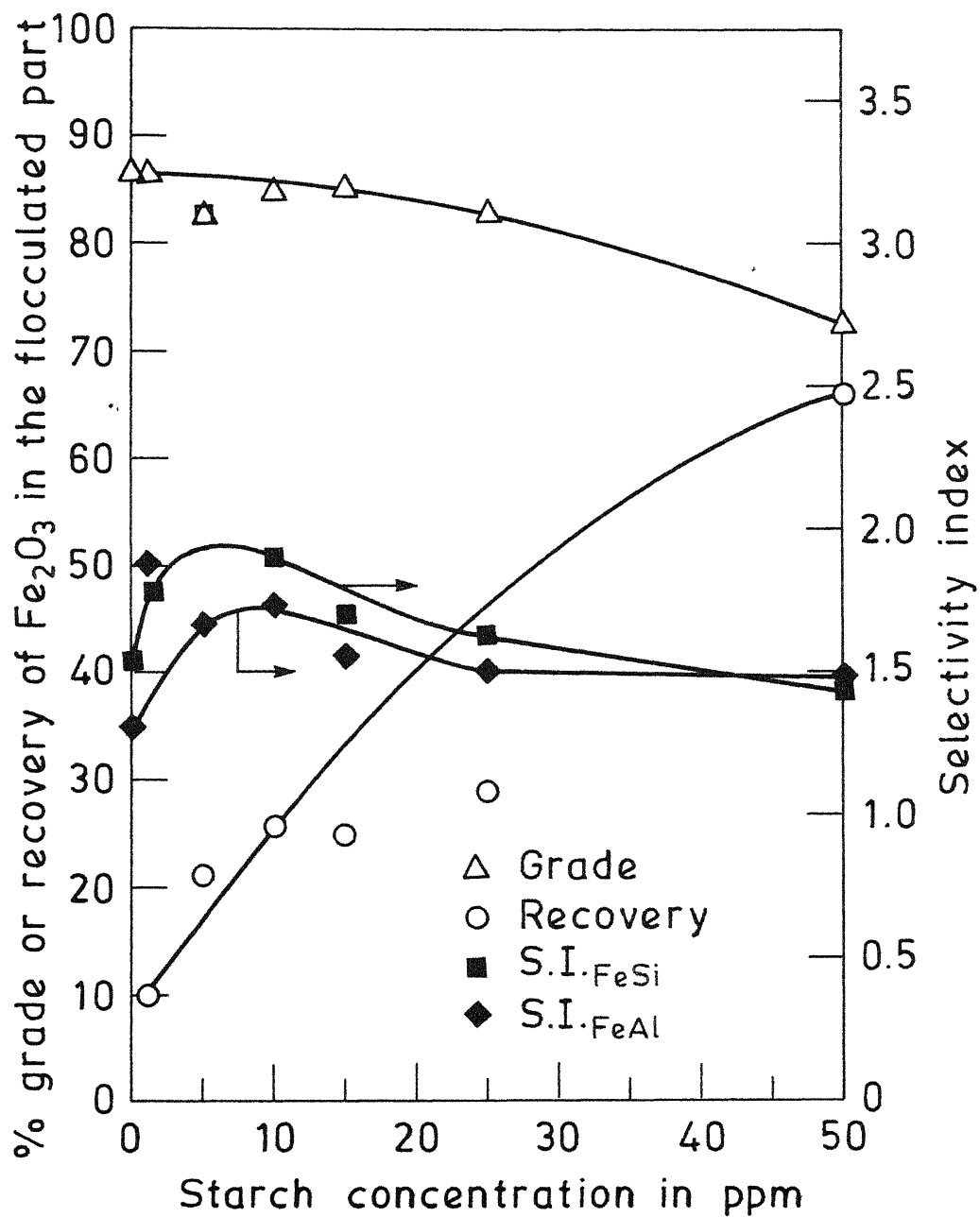
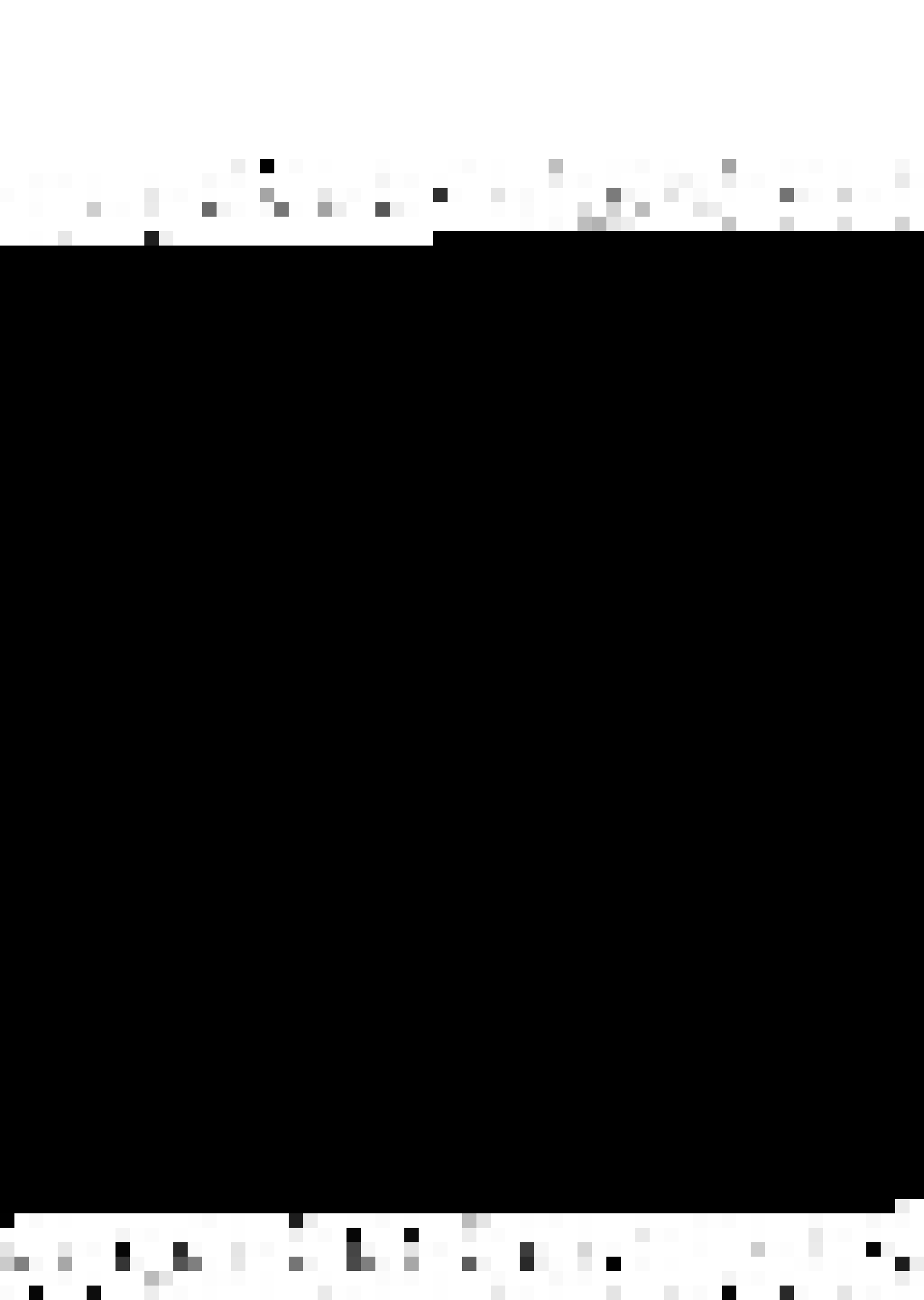


Fig. 20 - Effect of commercial starch concentration on flocculation of (1-20 μm) Barsua slime.
Ref. Table 452



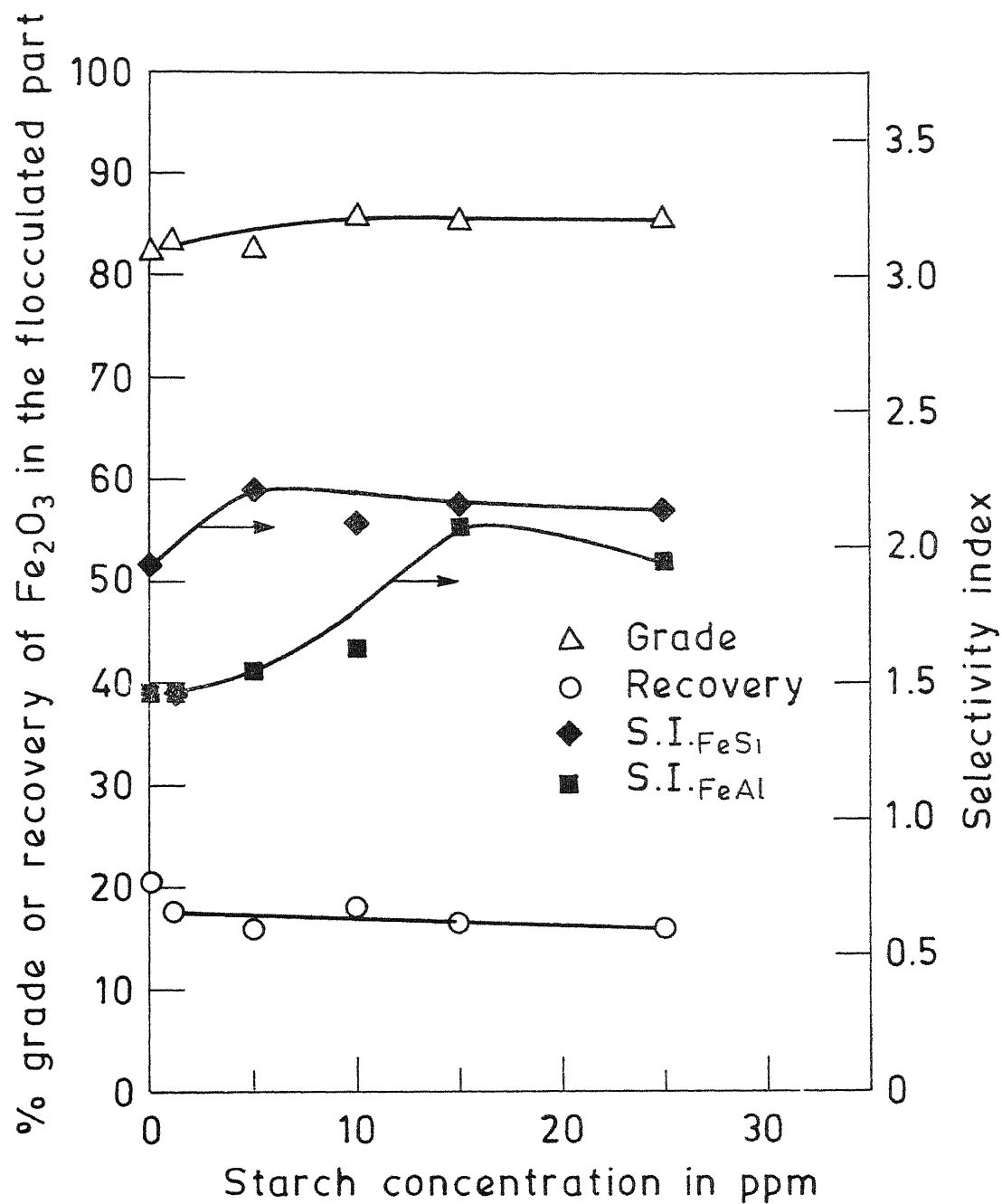


Fig. 30 - Effect of starch phosphate concentration on the flocculation of (1-²⁰₈ μm) Barsua slime.

Ref. Table 463



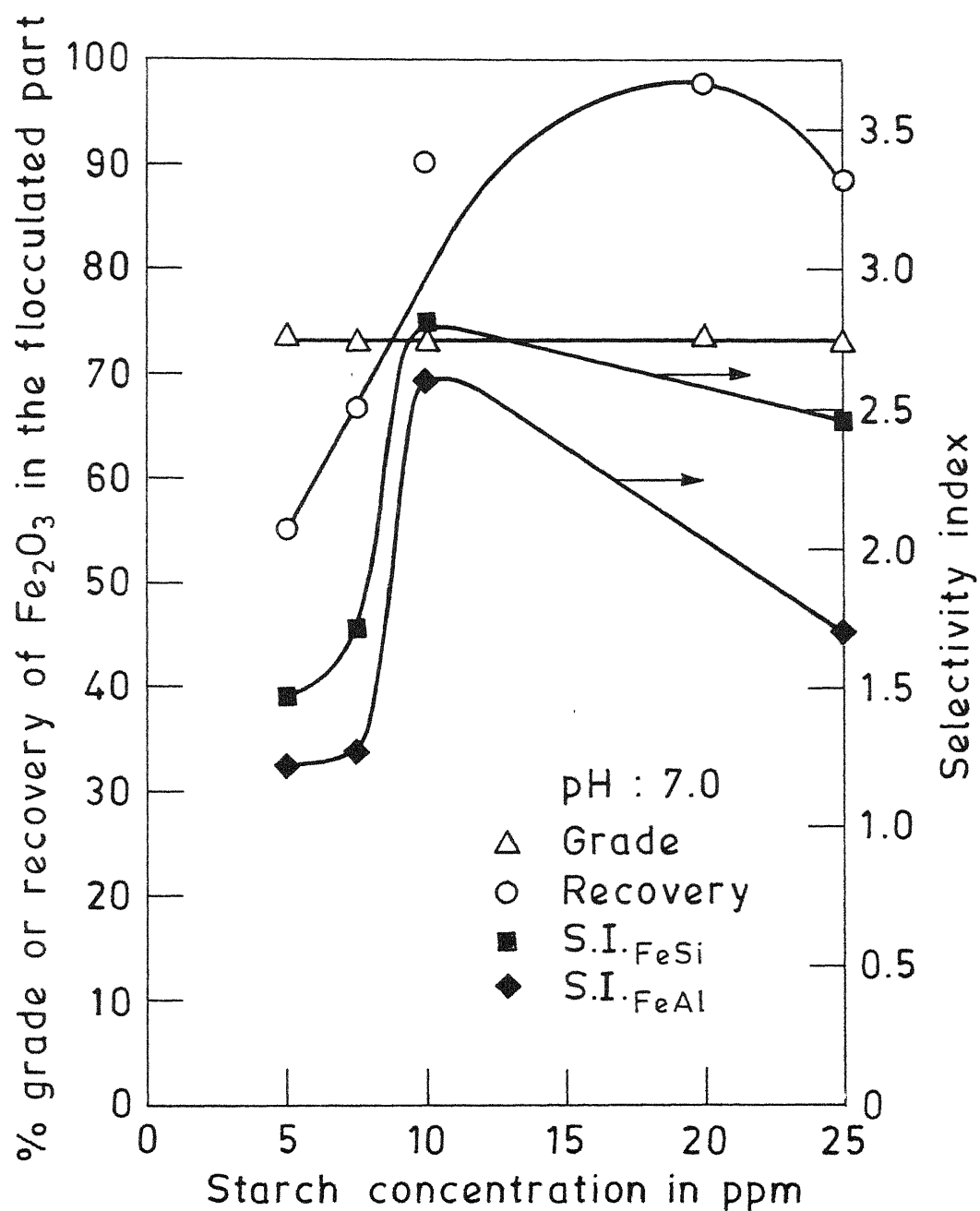


Fig. 84- Effect of starch concentration on the flocculation of (1-8 μ) Barsua slime, pH: 7.0
Ref. Table 507

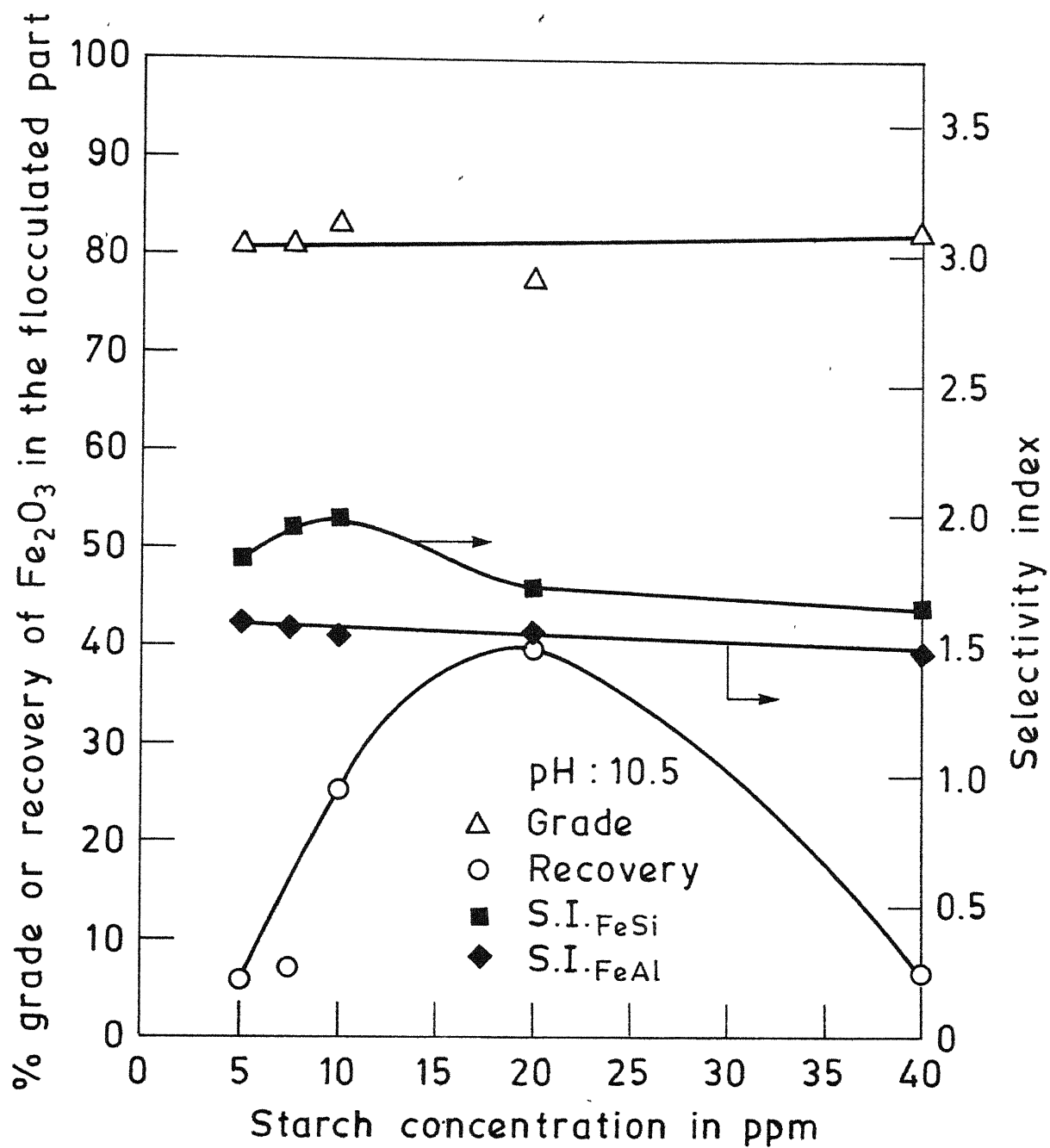


Fig. 5 - Effect of starch concentration on the flocculation of (1-8 μ) Barsua slime
pH : 10.5
Ref. Table 8



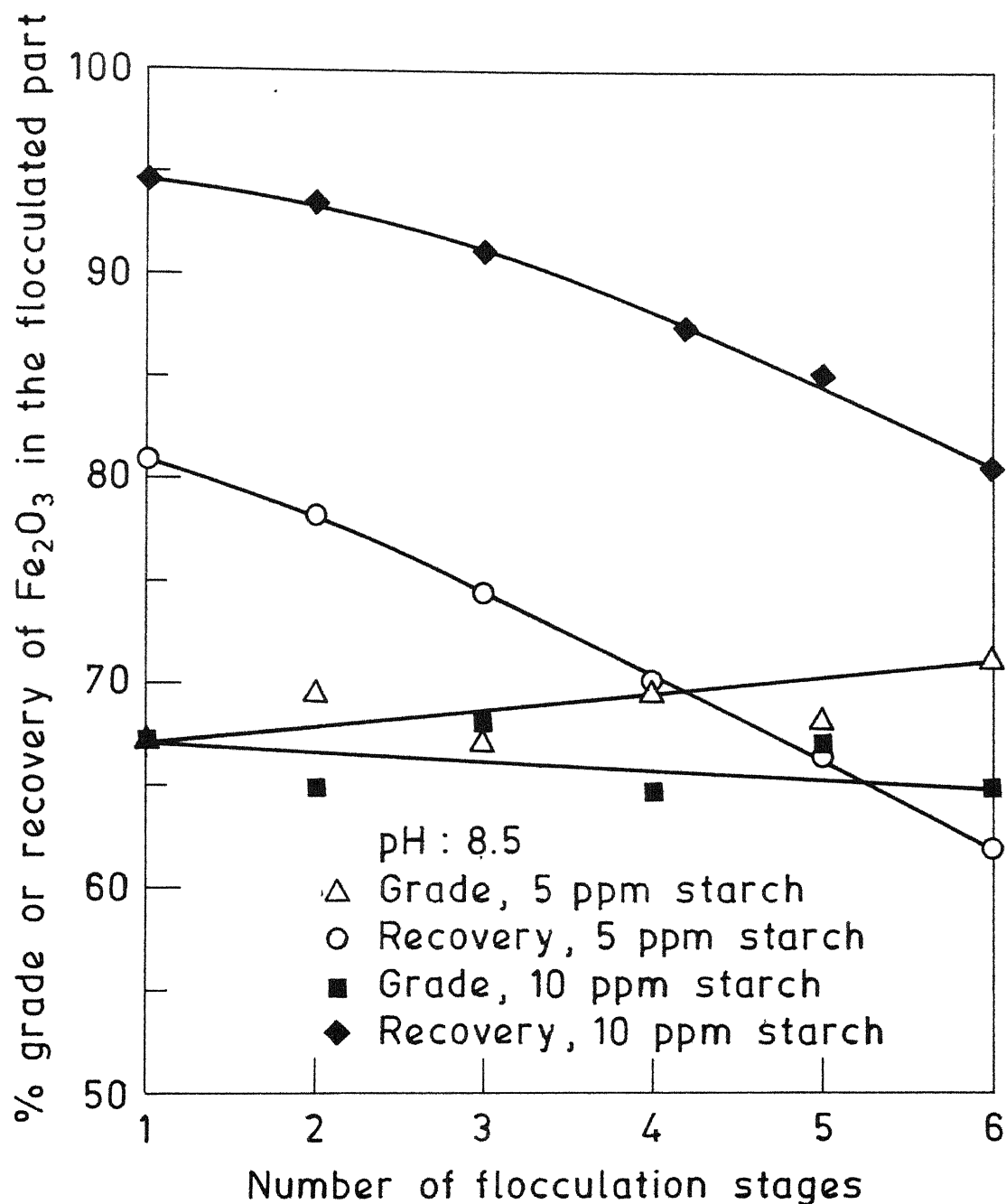


Fig. 633 - Effect of number of stages on the flocculation of 1-8 μ m Barsua slime by MCH starch at 8.5 pH.
Ref. Table 91 & 510



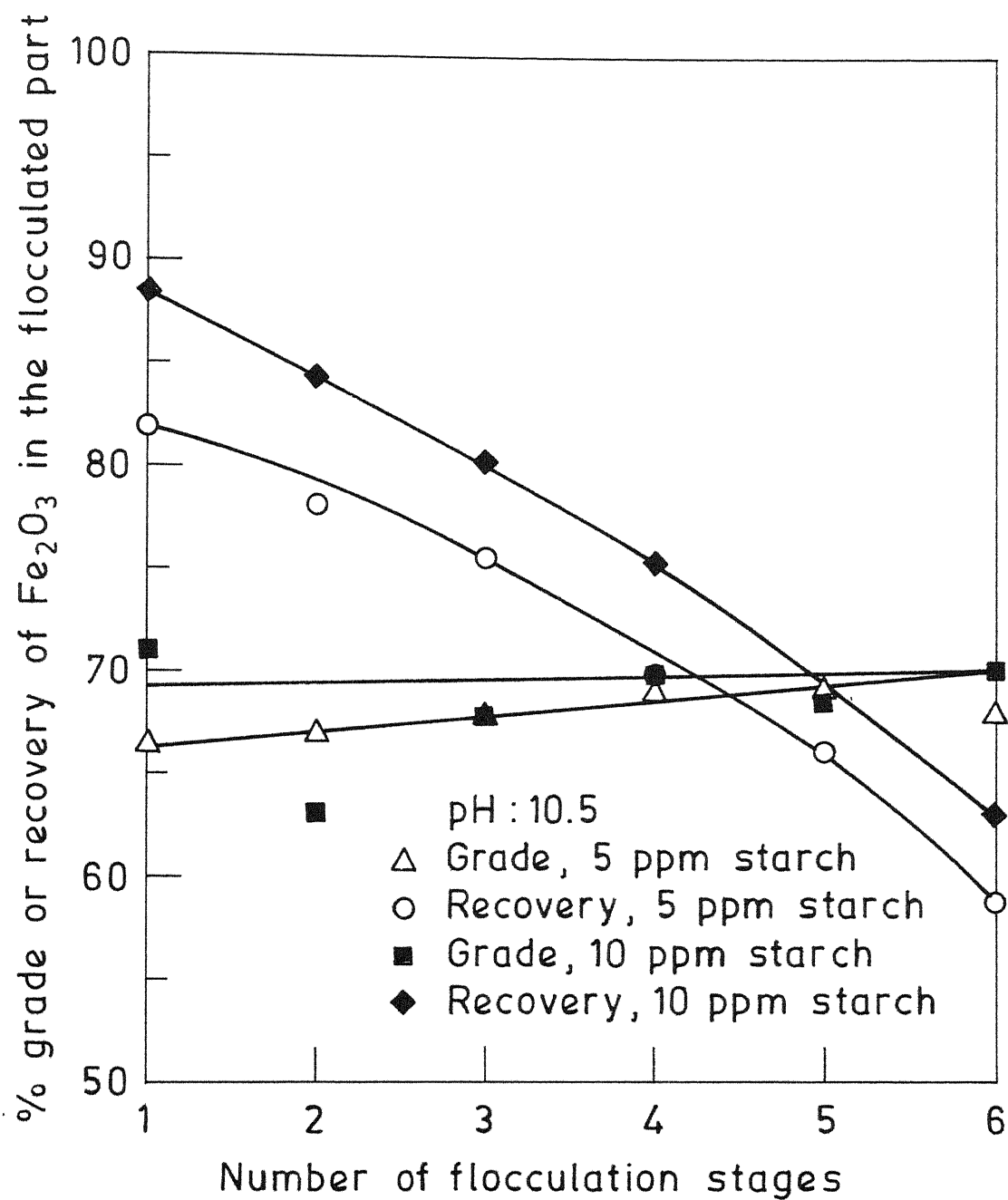


Fig. 734 - Effect of number of stages on the flocculation of 1-8 μm Barsua slime by MCH starch at 10.5 pH.
Ref. Table 5411&1255

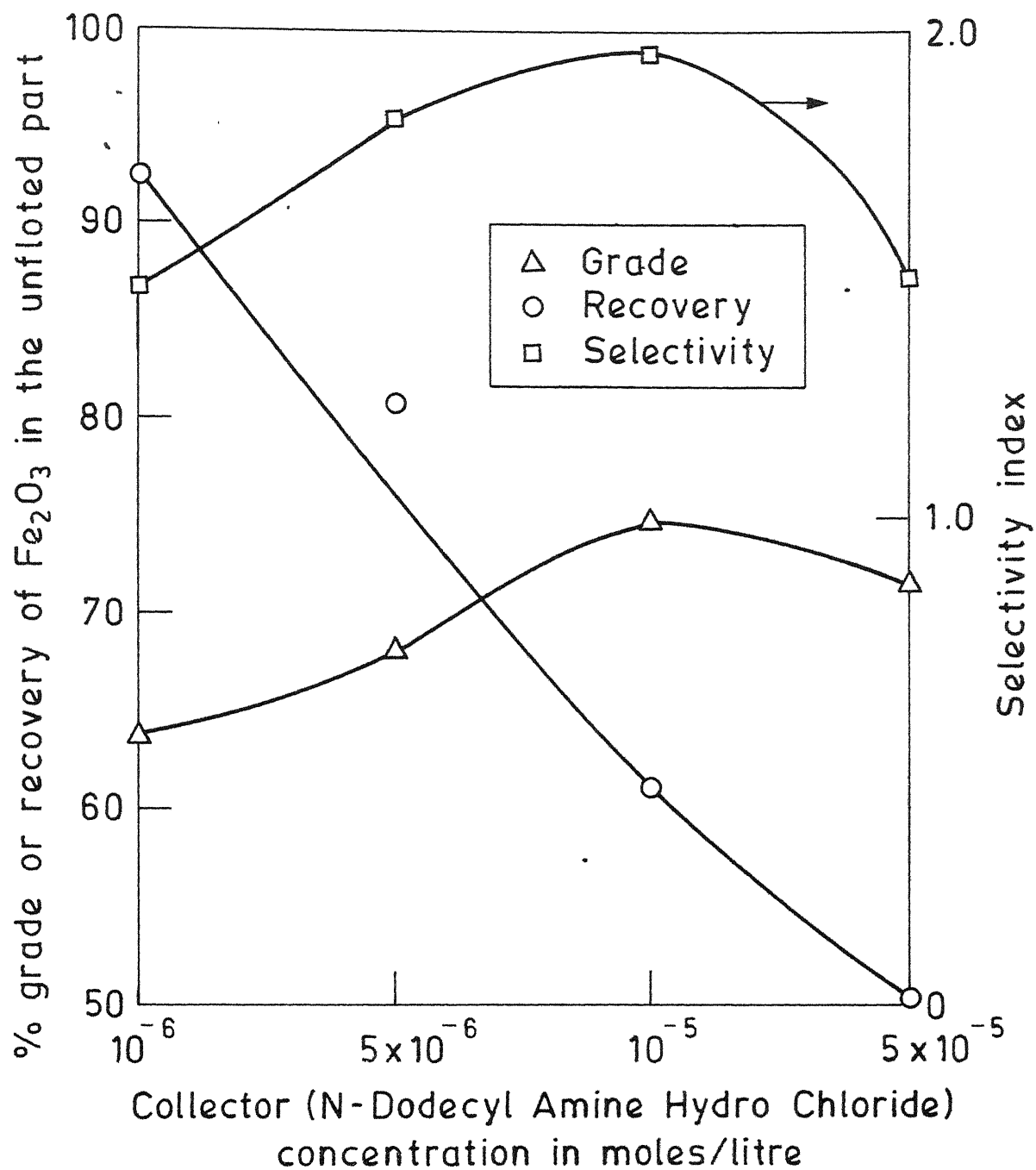


Fig. 8 - Effect of collector concentration on the flotation of 1 - 8 μm synthetic mixture of hematite, illite, kaolinite and montmorillonite.
Ref. Table 18

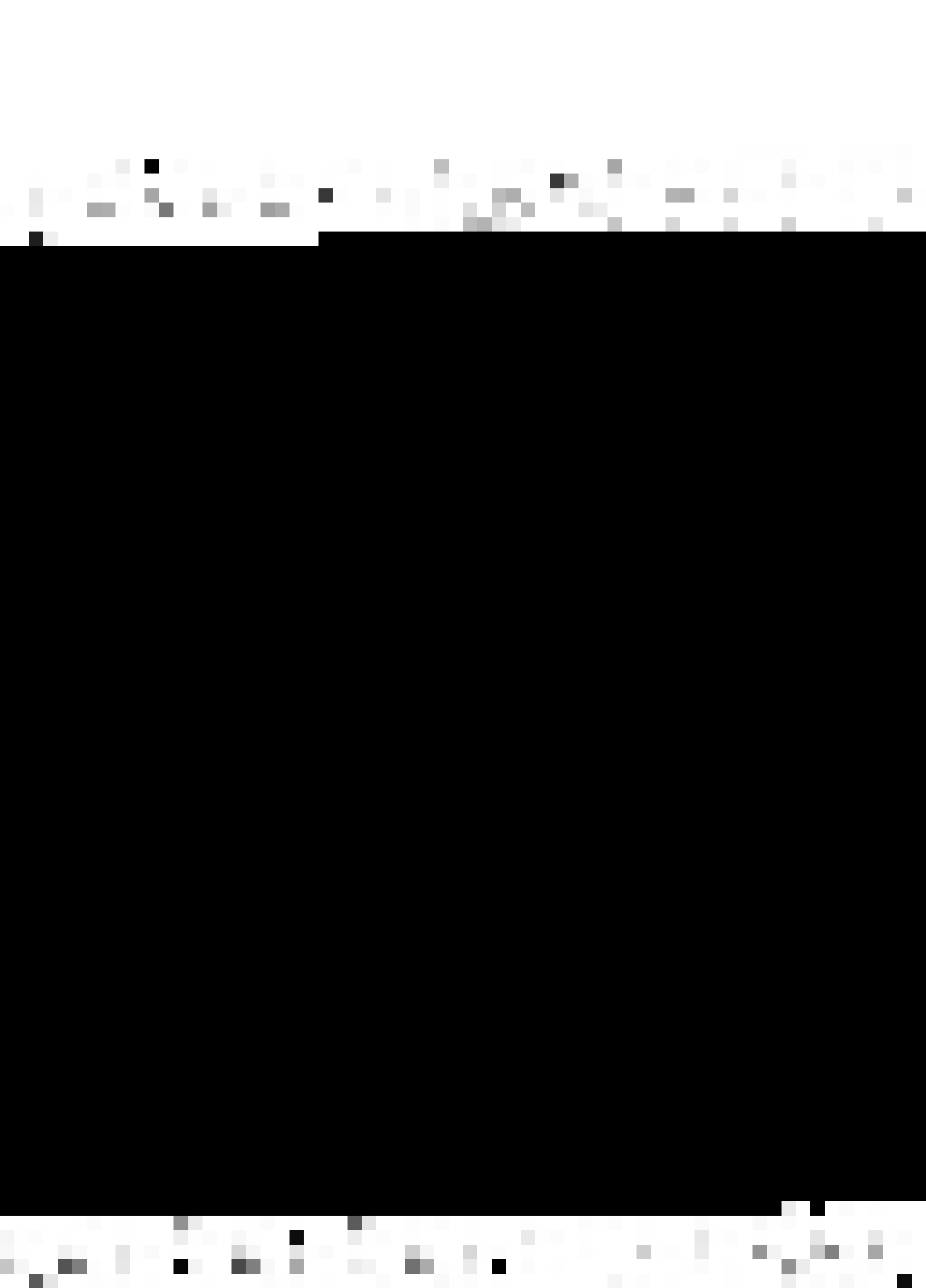


TABLE 47

Effect of Various Parameters on the Percentage
of (1-8) μ m fraction Flocculated (Single Stage)

Expt. No.	Starch concn. (ppm)	pH	Pulp density pct.	Stirrer speed (RPM)	Dispersant concn. (ppm)	pct. flocculated
1	200	6.7	1	2000	0	27.08
2	200	6.7	1	1000	0	75.07
3	300	6.7	1	1000	0	64.0
4	400	6.7	1	1000	0	46.3
5	200	6.7	2	1000	0	93.38
6	200	6.7	4	1000	0	97.13+
7	200	10.0	1	1000	0	50.5
8	200	6.7	1	1000	100	19.31
9 ^φ	200	6.7	1	1000	0	71.38
10 ^{φφ}	200	6.7	1	1000	0	76.9

φ Starch solution was prepared by usual procedure. For experiments no. 1-8 and 10 the stirring step was eliminated.

φφ Conditioning time 2 minutes, for all other experiments it is 1 minute.

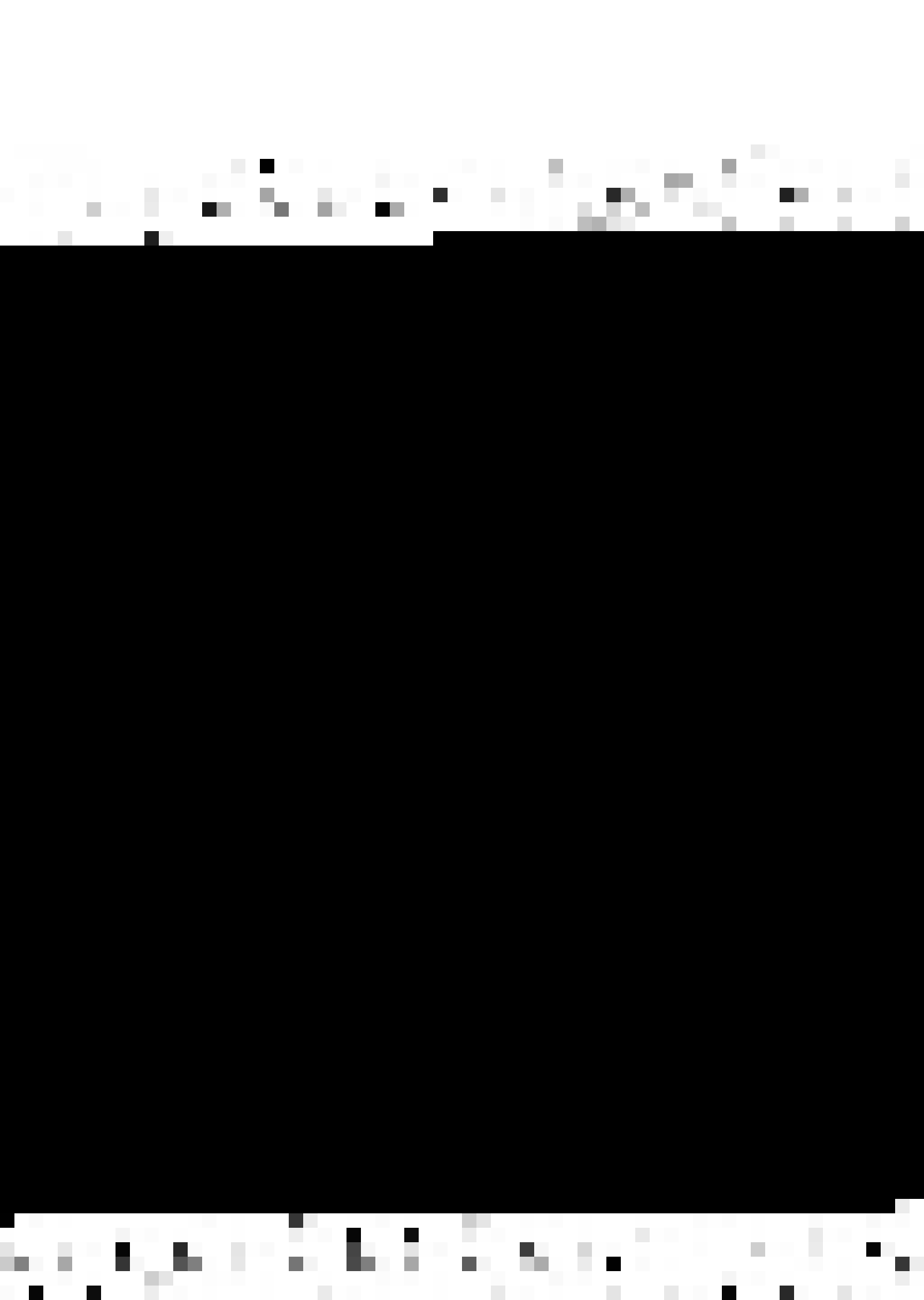


TABLE 496

Effect of Starch Concentration on two
stage flocculation of 1-8 μ m Barsua Slime

Pulp density 2 pct., pH 7.0 and 0 ppm dispersant

No.	Starch concentration (ppm)	Pct. Flocculated (Fraction A + B)
1	10	91.35
2	25	94.02
3	50	93.81
4	100	80.29
5	200	78.3
6	300	58.75
7	400	53.22

TABLE 52 70 9

Effect of Six Stage Flocculation on 1-8 μ m Barsua
Slime^φ (All previous data pertained to slime prepared from Lot I whereas data for this and following tables pertain to slimes prepared from Lot II received in suspended pulp form)

pH 8.5, Pulp density 2.0 pct, Na_2SiO_3 0 ppm.
NaF 50 ppm, Starch 5 ppm.

No. of stage	pct. Recovery	Chemical Analysis		
		pct. Fe_2O_3	pct. SiO_2	pct. Al_2O_3
1	80.95	67.2	10.88	16.80
2	78.03	69.6	11.48	15.84
3	74.37	67.2	11.68	14.52
4	70.07	69.6	11.70	14.00
5	66.53	68.0	9.64	15.88
6	61.68	71.2	7.58	16.00

φ Chemical Analysis: Fe_2O_3 66.4 pct., SiO_2 10.44 pct.,
 Al_2O_3 16.00 pct.



TABLE 53 10

Effect of Six Stage Flocculation on 1-8 μ m Barsua
Slime^φ

pH 8.5, Pulp density 2.0 pct., Na_2SiO_3 0 ppm,

NaF 50 ppm, Starch 10 ppm

No. of stage	pct. Recovery	Chemical Analysis		
		pct. Fe_2O_3	pct. SiO_2	pct. Al_2O_3
1	94.51	67.2	11.84	18.32
2	93.40	64.8	11.60	17.52
3	91.06	68.0	11.2	20.88
4	87.35	64.3	12.0	15.44
5	85.12	67.2	11.80	18.32
6	80.52	64.8	11.20	20.38

φ Chemical Analysis: Fe_2O_3 66.4 pct., SiO_2 10.44 pct.,
 Al_2O_3 16.00 pct.

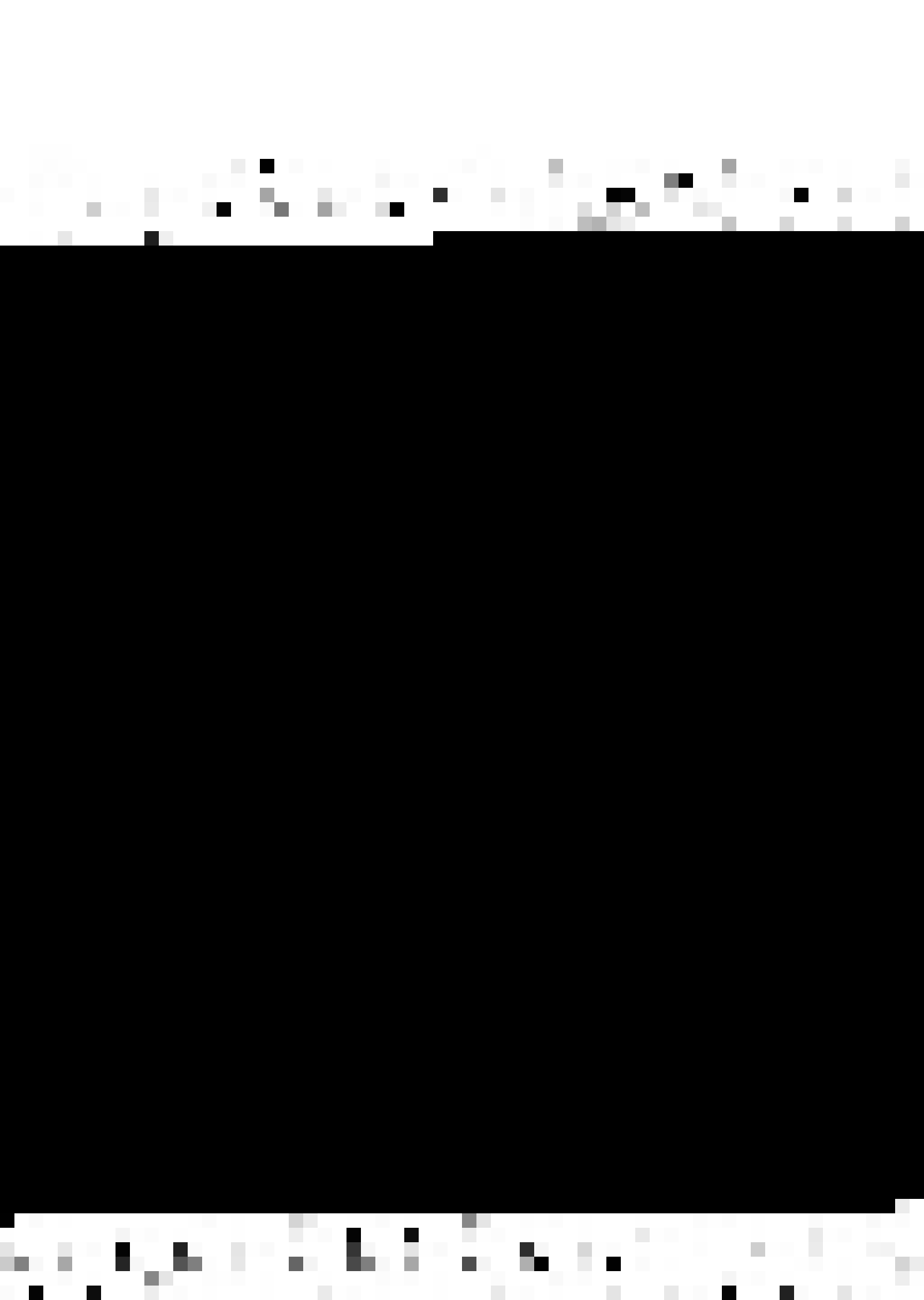


TABLE 5412

Effect of Six Stage Flocculation on 1-8 μ m Barsua Slime^φ
 pH 10.5, Pulp density 2.0 pct., Na_2SiO_3 0 ppm,
 NaF 50 ppm, Starch 5 ppm.

No. of stage	pct. Recovery	Chemical Analysis		
		pct. Fe_2O_3	pct. SiO_2	pct. Al_2O_3
1	81.94	66.4	9.70	15.76
2	78.0	67.2	11.0	15.28
3	73.55	68.0	10.0	15.88
4	69.65	69.0	10.0	15.32
5	63.61	68.8	10.1	16.48
6	58.76	68.0	10.12	16.40

φ Chemical Analysis: Fe_2O_3 66.4 pct., SiO_2 10.44 pct.,
 Al_2O_3 16.00 pct.

TABLE 55.2

Effect of Six Stage Flocculation on 1-8 μ m Barua Slime^φ
 pH 10.5, Pulp density 2.0 pct., Na_2SiO_3 0 ppm, NaF 50 ppm,
 Starch 10 ppm.

No. of stage	pct. Recovery	Chemical Analysis		
		pct. Fe_2O_3	pct. SiO_2	pct. Al_2O_3
1	88.56	71.23	11.06	14.48
2	84.22	63.2	10.56	13.20
3	80.27	68.0	10.24	13.60
4	75.26	69.6	10.64	13.20
5	71.90	63.3	9.56	16.40
6	62.91	70.4	9.14	12.00

φ Chemical Analysis: Fe_2O_3 66.4 pct., SiO_2 10.44 pct.,
 Al_2O_3 16.00 pct.

TABLE 55 13

Effect of Sodium Silicate on the Flocculation Behaviour
of 0-74 μ m Bhilai Ore

pH 7.0, Pulp density 1.0 pct., NaF 2 ppm, Starch 160 ppm

No.	Sodium Silicate concentration (ppm)	pct. Recovery (Fraction A and B)
1	5	70
2	15	67.49
3	25	64.9
4	50	61.5
5	100	61.35

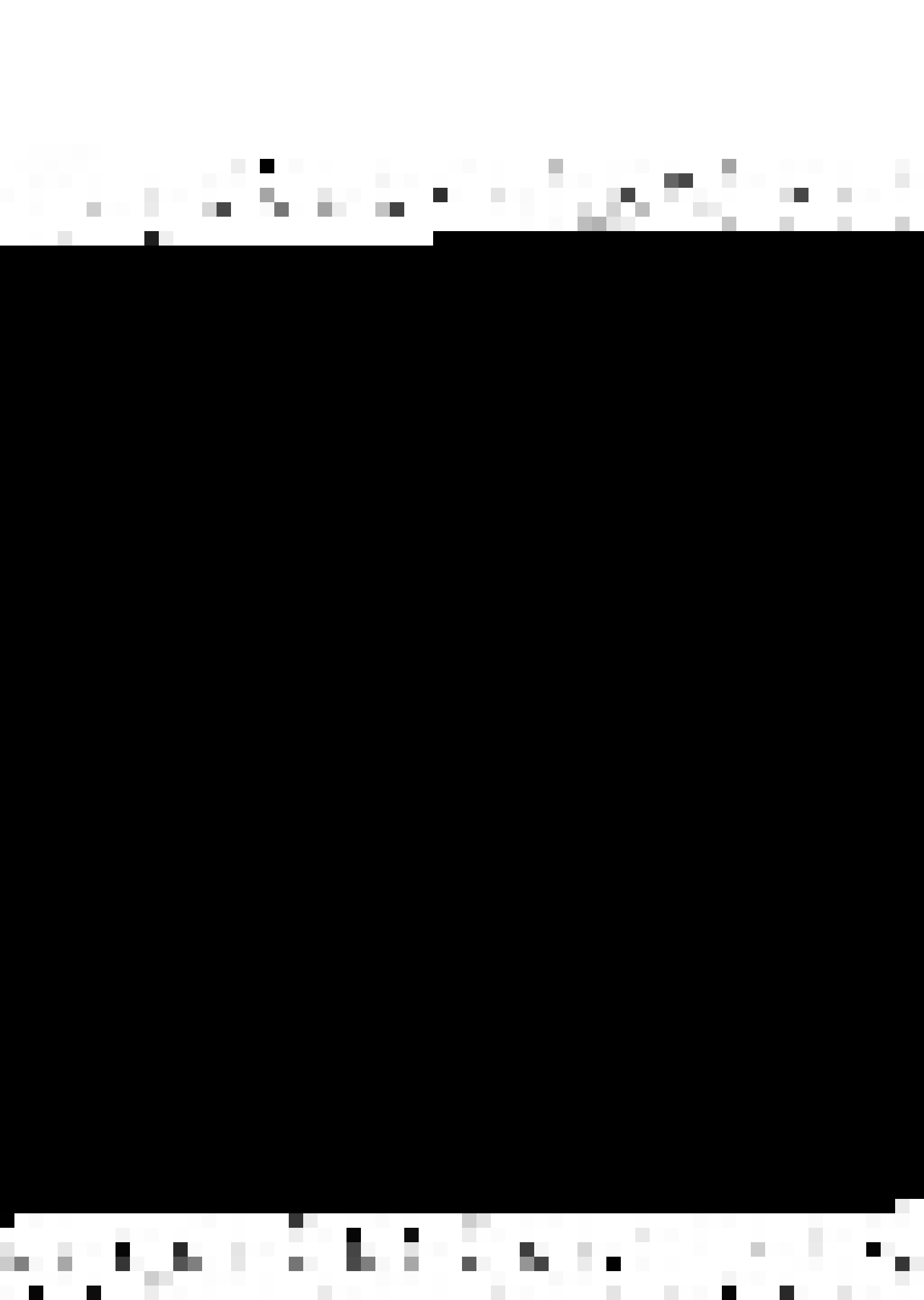


TABLE 59 16

Effect of Cationic Magnafloc^φ 292 Flocculant on
Flocculation of 1-8 μ m Parsua Slime⁺

pH 8.5, Pulp density 1.0 pct.

No.	Flocculant concentration (ppm)	pct. Recovery
1	0	29
2	0.5	47
3	1	53
4	2.5	83
5	5	100

φ Details given in reference no.19.

+ Test Performed in Small Cylinders.

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